with He gas + Air
Normal Amp Signal 245–345

HP 5890A GAS CHROMATOGRAPH

Accessory 19256A Flame Photometric Detector (FPD)
(HP 5890A Option 240)

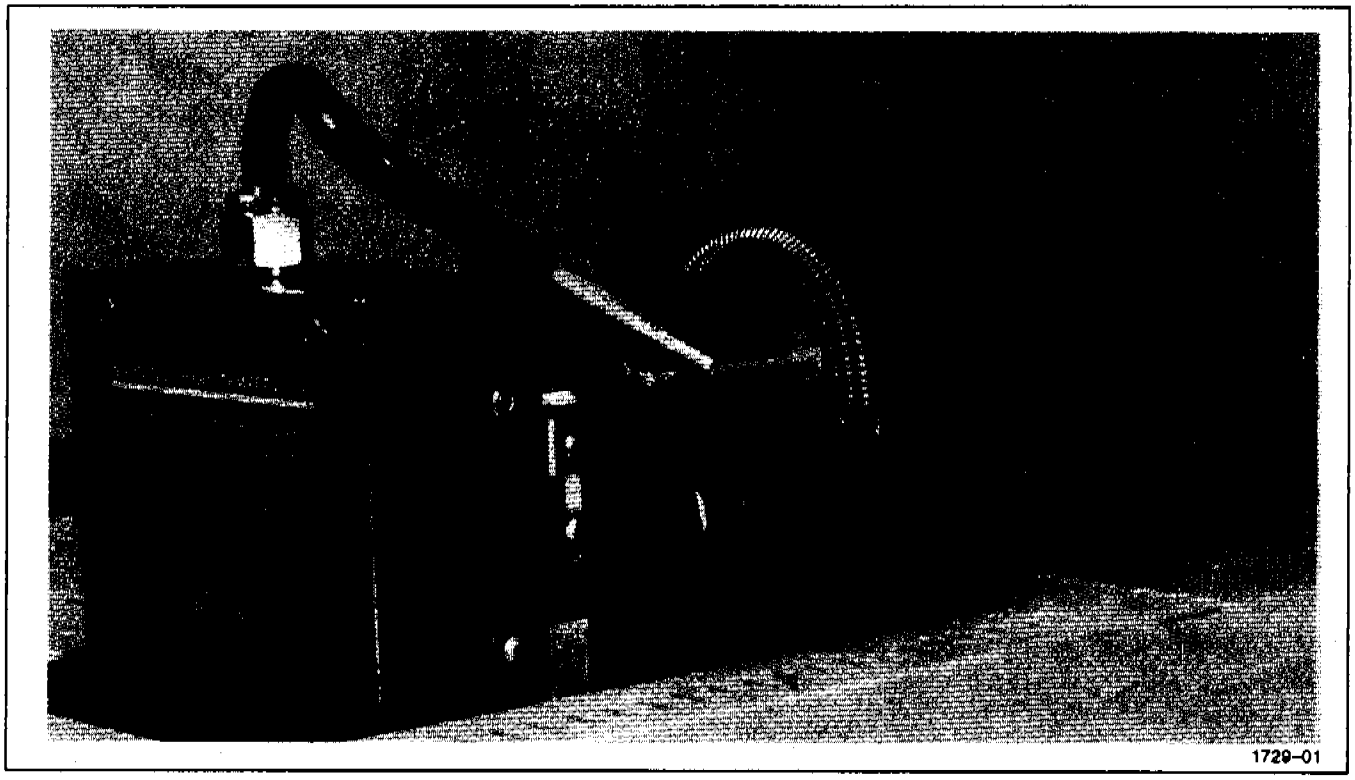


Figure 1. Flame Photometric Detector(FPD) on an HP 5890A GC

INTRODUCTION

SCOPE

This document provides installation, operating instructions, troubleshooting and service information for Flame Photometric Detectors (FPD) used on the HP 5890A Gas Chromatograph.

The "Accessories Manual" included with the detector provides Warranty, Safety and general information applicable to the installation of ALL accessories compatible with an HP 5890A Gas Chromatograph.

The supplied documents should be placed in Section 17 of the GC Reference Manual as a permanent addition to the manual. (Section 17 is specifically reserved for documents supplied with accessories).

Licensed under U.S. Patent No. 3,489,498.

Part No.
19256-90100

(11/86) Printed in U.S.A.

CONTENTS

CONTENTS (CONTINUED)

ENVIRONMENTAL CONDITIONS

The FPD is designed to operate in a Class B Industrial/Commercial environment (i.e. temperature 0 to 55 degrees C; relative humidity 5 to 95%). This is identical to the environment specified for the HP 5890A GC as described in Section 2 Volume 1 of the HP 5890A GC Reference Manual (Part No. 05890-90110).

NOTE

If the FPD is installed on an instrument that is not located in such an environment (or better), it may not be possible to meet the published FPD specifications.

DESCRIPTION

THEORY OF OPERATION, ABSTRACT

The FPD selectively detects phosphorus or sulfur-containing compounds by burning the column effluent in a tuned flame and measuring a selected spectral portion of the emission above the flame. Light emission at a wavelength characteristic of the molecular species selected passes through an optical filter to a photomultiplier tube (PMT) where it is converted to a current and amplified. This current is then further amplified and converted to a millivolt output by the HP 5890A electronics.

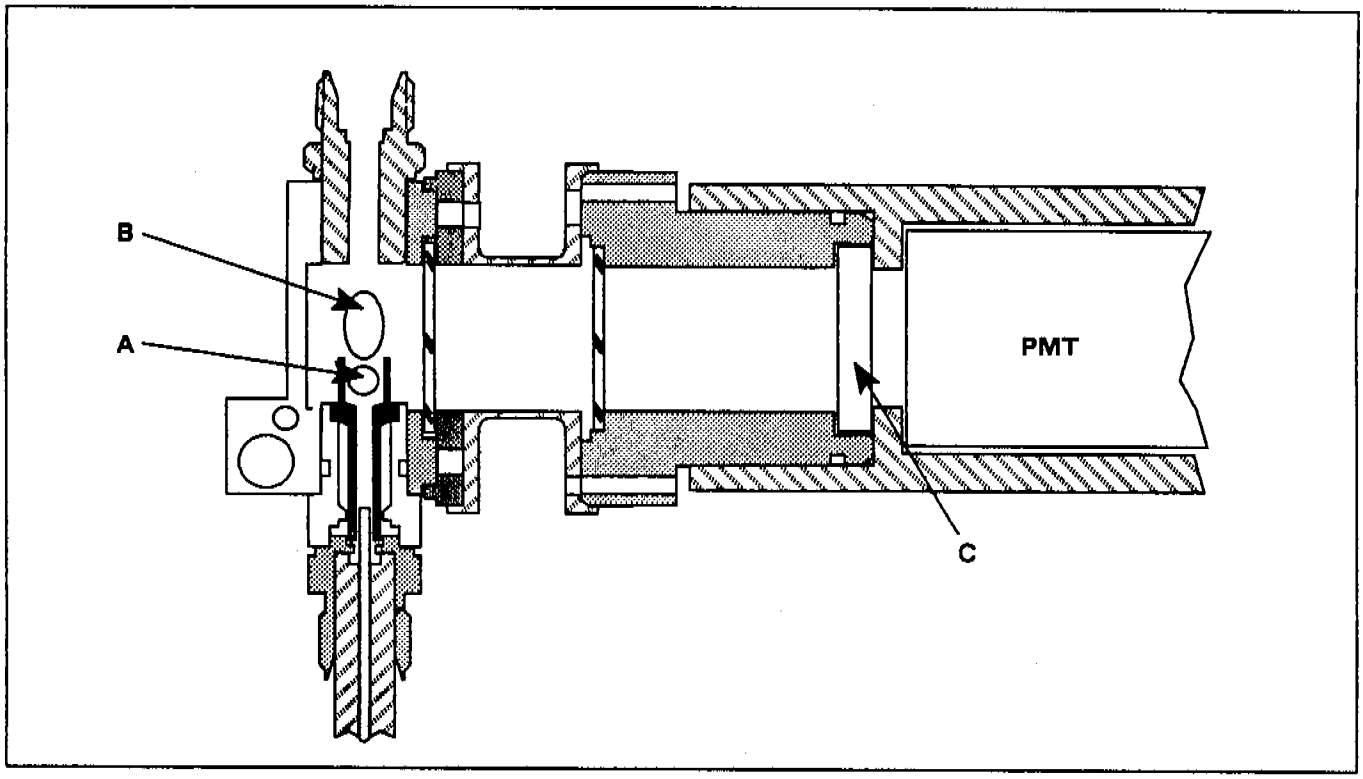


Figure AA. Sectional View of the HP 5890A GC Flame Photometric Detector

THEORY OF OPERATION, DETAILED (FIGURE AA)

Basic operation of the Flame Photometric Detector (FPD) is as follows:

The sample is burned and the heteroatom species is activated in the flame at position A in the reference figure (Figure AA).

The detector flows move the activated species to position B (Figure AA) above the flame where it decays and gives off a band of light that is unique to the species being measured.

The light passes through the proper narrow band-pass optical filter (position C) that was chosen for the species being measured. All other light that might be given off from the flame is blocked by this optical filter.

The light then strikes the photo-reactive coating on the photomultiplier tube (PMT). At this point the light photon knocks loose an electron.

The electron is then accelerated to the grid and first dynode of the PMT by the voltage differential. On striking the surface of the dynode the electron knocks loose several other electrons and these are then accelerated toward the second dynode, etc. There are several dynodes inside the PMT and the total interaction gives rise to an overall multiplication of up to a million-fold, depending on the total voltage applied. Higher voltages result in greater current amplification but may also result in higher background noise levels.

The current from the PMT is then further amplified and digitized by the HP 5890A FPD electronics board. This digitized signal is then processed by the HP 5890A GC electronics board either as a digital signal on the communications output or as a voltage signal on the analog signal output.

There are several conditions that must be satisfied and "optimized" for the detector to function properly. Some of these are:

Flame

Most obvious, of course, the flame must be lit. Less obvious however is that the flame must provide both the proper temperature and chemical conditions necessary to "burn" the molecule injected, as well generate and activate the chemical species that will utimately decay to emit the photon of light unique to that species.

Also this emission cannot occur in the flame itself since the PMT cannot "see" light generated in the flame. The PMT is shielded from the flame emission by the design of the flame cup.

Because of these considerations, it is important that the gas flows to the detector be set properly for check-out. The gas flows may need to be optimized for operation with the samples of interest.

Filters

The proper filter must be selected to allow the wave length of light that will be emitted to pass through to the PMT. An incorrect filter selection will result in a low signal - or no signal at all.

The optical filters provided pass light of either 535 nanometers (5250 Angstroms) wavelength for the phosphorus mode or 393 nanometers (3930 Angstroms) wavelength for the sulfur mode filter.

Photomultiplier Tube

The operational requirements for the PMT are:

(1) A high voltage must be applied to the terminals (this is preset to 850 VDC) and (2) the "dark current" from the PMT must be below some typical value. The "dark current" is equated to the "flame-off" noise level of the FPD.

The "flame-off" noise can be determined by measuring the signal output noise level with the PMT connected to the electronics, the detector "ON" and the flame "OFF". Compare this noise level to the "electronics noise" below.

Electrometer and Power Supply Board

The electronics board serves two functions: (1) to provide the -850 VDC to the PMT and (2) to amplify the current provided by the PMT.

The main requirement for operation of the electronics board is that it must operate at a low enough signal noise level to allow the the desired MDL to be obtained. For a new instrument, this is equated to the electronics noise of the detector. This can be determined by plotting the signal noise with the PMT disconnected. Calibrating the output from the electronics board cannot be done by the user.

Light Emission

A detailed discussion of the reactions that occur in the reaction zone of the detector is beyond the scope of this manual but is contained in documents referenced here.

It should be noted that the chemistry occurring in the flame dictates that the light emitted cannot be linear with sulfur concentration, but that the light is approximately proportional to the square of the sulfur atom concentration. (Hewlett-Packard Integrators, work stations and computer systems are available to do multi-level calibration of this detector.)

The process involved in the molecular emission of light from sulfur containing compounds are described by the following reactions.

SULFUR COMPOUND burned to form H_2S

$$H_2S + H \underset{}{\overset{K_2}{\rightleftharpoons}} HS + H_2$$

$$HS + H \overset{K_3}{\rightleftharpoons} S + H_2$$

$$2S + M \overset{K_4}{\rightleftharpoons} S_2^* + M$$

$$S_2^* \dashrightarrow S_2 + h\nu$$

Where M represents a species present in the flame which participates in the process without undergoing any change itself. On the basis of these equations it can be concluded that the chemiluminescence of S2 is associated with the recombination of two sulfur atoms followed by a three body collision which results in the release of light.

The species leading to the response in the phosphorus mode has been proposed to be an HPO species. The final reaction being

$$HPO^* \text{ ---> } HPO + h\nu$$

QUENCHING

One problem that can be serious with the FPD is the light absorption that can occur in the flame. This can happen in two different ways. The first is the "hydrocarbon quenching" that occurs when there is a high concentration of carbon dioxide in the flame at the same time as the sulfur atom species. In this case the light that is emitted by the sulfur species is absorbed in position B by some type of CO2 species. The CO2 would exist in the flame at a relatively high concentration if a hydrocarbon peak were eluting at the same time as the sulfur compound.

The second quenching is "self-quenching" that can occur at high concentrations of the hetro-atom species. In this case some other ground state (unactivated) species could re-adsorbe the photon that had been previously emitted, effectively preventing it from reaching the PMT.

The overall effect of the inter-related reactions in the FPD means that good chromatographic practices should be used. The column should provide good separation of the compounds, those that contain sulfur or phosphorus as well as those that do not but may absorb light. A careful, multi-level calibration is well worth the investment!

Detector and gas cleanliness should be maintained in order to have consistant responses. Since most sulfur and phosphorus compounds contain very active sites the injection and column systems should be kept very clean.

References

1. Burnett, C.H., Adams, D.F., and Farwell, S.O., "Relative FPD Responses for a Systematic Group of Sulfur-Containing Compounds", **J. Chrom. Sci.**, 16 (1978), pp 68-73.

2. de Souza, T.L.C., Lane, D.C., and Bhatia, S.P., "Analysis of Sulfur-Containing Gases by Gas-Solid Chromatography on a Specially Trated Porapak QS Column", **Anal. Chem.**, 47:3 (1975), pp 543-545.

3. McGaughey, J.F. and Gangwal, S.K., "Comparison of Three Commercially Available Gas Chromatographic Flame Photometric Detectors in the Sulfur Mode", **Anal. Chem.**, 52:13 (1980), pp 2079-2083.

4. Pearson, C.D., "The Determination of Trace Mercaptans and Sulfides in Natural Gas by a Gas Chromatography-Flame Photometric Detector Technique", **J. Chrom. Sci.**, 14 (1976), pp 154-158.

5. Sugiyama, T., Susuki, Y., and Takeuchi, T., "Intensity Characteristics of S=S Emission for Sulfur Compounds with Flame Photometric Detector", **J. Chrom. Sci.**, 11 (1973), pp 639-641.

DETECTOR/HEATED ZONE MODULE

The detector/heated zone module consists of the transfer line, column adapters, heated zone, burning chamber, detector mounting hardware and the PMT housing.

This assembly is mounted on the HP 5890A GC detector "B" location with the detector block extending over the top cover of the instrument. Only a single FPD can be installed on the HP 5890A GC and it must be installed in the "B" detector location.

Detector temperature is regulated by the "B" detector heated zone controls of the HP 5890A GC. The recommended operating temperature is from 120 degrees C minimum to 300 degrees C maximum. Continuous operation at temperatures above 250 degrees C will reduce the detector seal life.

Optical Filters. Two optical filters are provided, one which transmits 393 nanometers wavelength for specific sulfur response and another which transmits 525 nanometers for specific phosphorus response. One of the filters must be installed in the light path between the detector block and the PMT, as explained in this document.

Fused Silica Liner. A major feature of the HP 5890A FPD is the use of an inert fused silica insert liner through the isothermal transfer line. This liner allows fused silica columns up to 530u ID to be run right to the base of the flame, minimizing sample tailing or loss on chemically active sites. The liner is completely compatible with all standard packed columns as well.

Detector Temperature Considerations. Operating the FPD at less than 150 degrees C in high humidity will cause water to condense and buildup in the block, reducing detector response and eventually causing flameout. If this occurs, raise the detector block temperature.

Operating the detector at temperatures above 250 degrees C may lead to some thermal decomposition or adsorption of many thermally labile phosphorus and sulfur compounds.

PNEUMATICS MODULE

The pneumatics module consists of the flow manifold block and tubing for control and delivery of (1) AIR or OXYGEN, (2) HYDROGEN and (3) Make-up Gas (NITROGEN) to the detector. Two modes of operation are available; the AIR MODE and the OXYGEN MODE – depending on the supply gas that was selected. General flow rates are shown in the following table for both modes.

The FPD flow manifold block occupies the B flow manifold block location on the HP 5890A GC.

General Flow Rates (ml/min.)	OXYGEN Mode	AIR Mode
OXYGEN	20	0
AIR	0	100
HYDROGEN	75	75
AUXILIARY NITROGEN PLUS CARRIER	140	30

NOTES

1. The OXYGEN MODE is the most versatile for flow optimization. It allows totally independent control of absolute and relative oxygen, hydrogen and nitrogen flows.

2. The AIR MODE has three worthwhile features:
   a. It has a safer oxidant supply,
   b. Is simpler to use than the Oxygen Mode,
   c. It has adequate sensitivity for most applications.

Use of Accessory 19246A Auxiliary Flow Panel. Because it is necessary to have precisely regulated gas flows to the FPD, it may be desirable to install an Accessory 19246A Auxiliary Flow Panel. This is particularly true when a number of instruments are connected in parallel to the same gas supplies where pressure and flow may fluctuate considerably. The flow panel includes three precision supply pressure regulators and gauges (figure 2).

For more information on this accessory, consult your Hewlett-Packard representative.

Use of Electronic Flow Sensor (EFS). The FPD is extremely flow sensitive, particularly to variations in hydrogen and air (or oxygen). One way to minimize the day-to-day changes in detector sensitivity is to plumb the hydrogen and air (or oxygen) through the two channels of the HP 5890A Electronic Flow Sensor (EFS).

The EFS may be user-calibrated for the air (or oxygen) channel by following the procedure described in Section 7 of the HP 5890A Reference Manual supplied with the gas chromatograph.

If plumbing air or oxygen through the EFS, the EFS should be set for "Nitrogen" gas to get a close approximation of flow. (The EFS Does not include an "Oxygen" setting.)

The HP 5890A EFS has excellent day-to-day reproducibility, allowing quick easy verification that the two most critical flows for FPD sensitivity are retained at desired values.

For instructions on how to plumb the FPD flows through the EFS, refer to figure 18.

For a description on how to use the EFS as a leak tester for the FPD, refer to the troubleshooting section of this document.

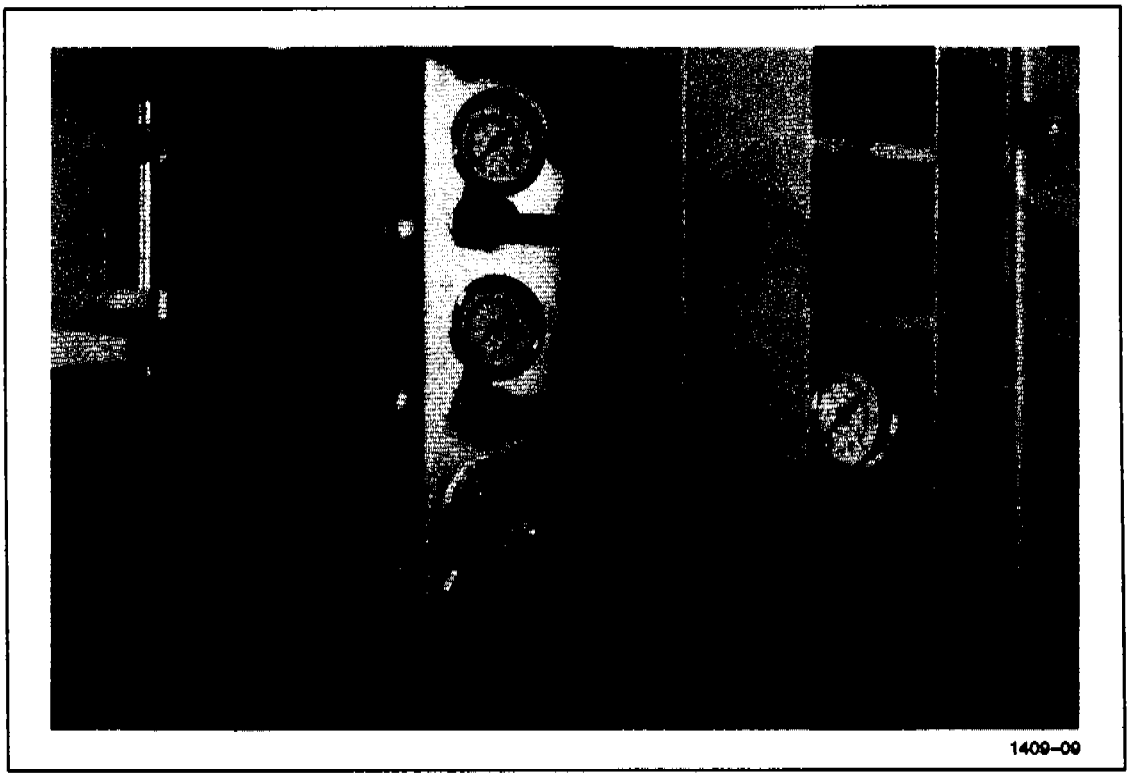


Figure 2. Auxiliary Flow Panel (Accessory 19246A) Installed

ELECTRONICS MODULE

The electronics module consists of the FPD printed circuit board, resistor network cable assembly and photomultiplier tube (PMT). The printed circuit board contains the electrometer, digitized signal output and the high voltage supply for the PMT.

The board mounts in the normal HP 5890A detector "B" electronics board slot on the right side of the mainframe.

INSTALLATION

GENERAL INFORMATION

Before beginning the following installation procedure, read the "Accessory Manual" included in the shipment. This guide contains information applicable to the installation of most accessories available for the HP 5890A Gas Chromatograph.

CAUTION

To avoid eye injury, always wear safety glasses or a face shield when working on an instrument. This is particularly important when using compressed gases and when installing columns.

INSTALLATION RESTRICTIONS

HP 7671/2A Automatic Samplers cannot be mounted on an HP 5890A GC when an FPD is installed due to mechanical interferences. An HP 7673A Automatic Sampler can be mounted when the FPD is installed.

The FPD requires the entire detector "B" location and any detector presently installed must be removed before an FPD can be added to the instrument. "Entire" means removing the detector, detector printed circuit board and the flow manifold block. After this is done, the FPD can be installed by following the instructions below.

PARTS SUPPLIED

Before proceeding with the installation, check the parts received with the parts listed and shown in the Parts Supplied list (blue pages) at the back of this document. If something is wrong, refer to the HP Warranty in the "Accessories Manual" for guidance.

SERIAL NUMBER LABEL

Peel the backing off the serial number label and apply it to the oven door threshold next to instrument serial number label (figure 3).

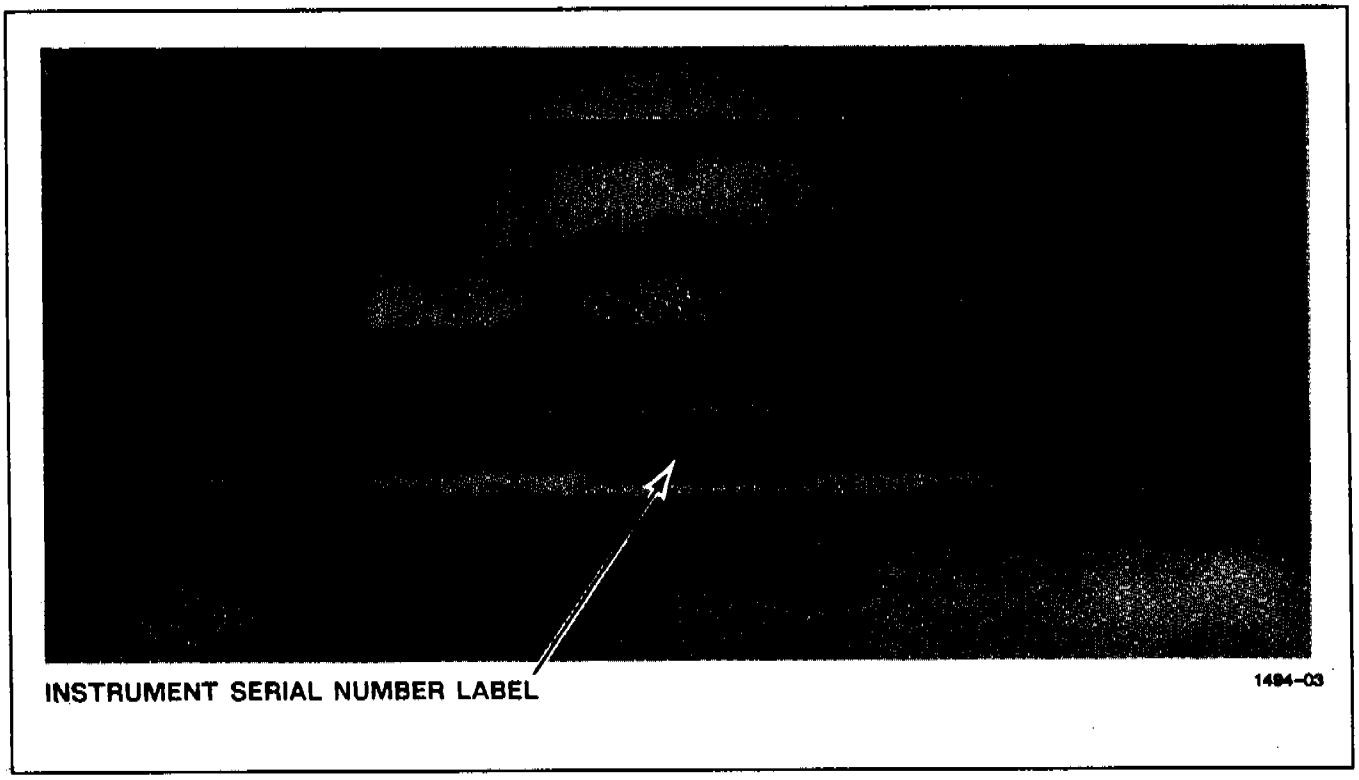


Figure 3. Location of Serial Number Label

TOOLS REQUIRED

The following tools, or their equivalent, are required to install an Accessory HP 19256A FPD on the HP 5890A Gas Chromatograph.

Pozidrive-type Screwdriver, No. 1 tip, 3-inch blade, P/N 8710-0899

Pozidrive-type Screwdriver, No. 2 tip, 3-1/2-inch blade, P/N 8710-0900

Nut-driver, 1/4-inch, P/N 8720-0002

Combination Wrench, 5/16 (0.312)-inch, P/N 8720-0015

Open End Wrench, 9/16 X 5/8 (0.562 X 0.625)-inch, P/N 8720-0010

INSTALLATION PROCEDURE

Turn off power to the GC and disconnect the main power cord. Allow the the instrument to cool, then turn off supply gases.

Remove the hinged top cover, the side panels and the rear panel. The top cover is replaced by the cover supplied but the plastic injection port pan is reused and must be transferred to the new top cover.

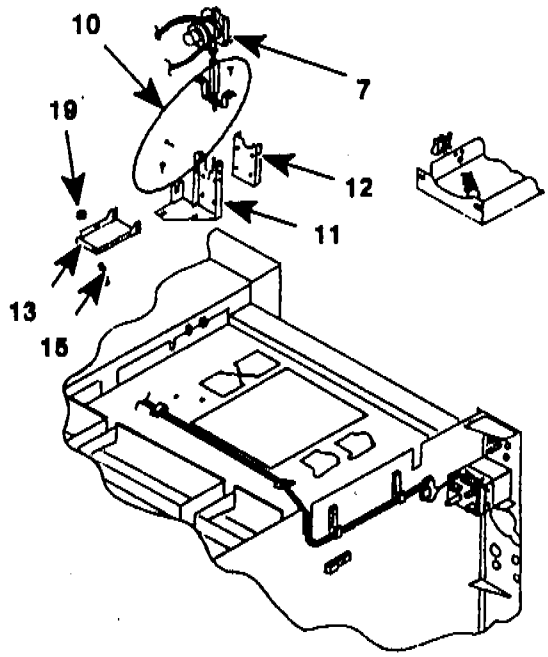
WARNING

HAZARDOUS VOLTAGES ARE PRESENT IN THE MAINFRAME WHEN THE INSTRUMENT POWER CORD IS CONNECTED. TO AVOID A POTENTIALLY DANGEROUS SHOCK HAZARD, THE INSTRUMENT SHOULD BE "OFF", AND THE POWER CORD DISCONNECTED, BEFORE REMOVING THE REAR PANEL.

CAUTION

AS INSTALLATION OF THIS ACCESSORY WILL NECESSARILY INVOLVE EXPOSURE TO REFRACTORY CERAMIC FIBER, IT IS ADVISED THAT 1) THE TOP OF THE GC BE VACUUMED BEFORE REMOVING THE BLANK COVER PLATE, 2) THE INSTALLER WEAR GLOVES (LIKE FIBERGLASS INSULATION, REFRACTORY CERAMIC FIBER CAN CAUSE IRRITATION); IN ADDITION, 3) A SIMPLE RESPIRATORY MASK MAY BE WORN TO PREVENT INHALING THE AIRBORNE FIBERS, AND AFTER DETECTOR INSTALLATION, 4) THE TOP OF THE GC AND THE OVEN AREA BE VACUUMED BEFORE TURNING THE POWER ON THE UNIT.

1. Remove the blank cover plate and the insulation below it from the detector "B" mounting location. Use a screwdriver or pencil through the detector column fitting opening in the oven liner to dislodge the pre-cut insulation plugs. Save the insulation for step 4.



Item	Description
1	PMT Assembly
2	Printed Circuit Board
3	Serial Number Label
4	Switch Cable Assembly
5	Hinged Top Cover
6	FPD Cover Assembly
7	Detector / Flow Assembly
8	Sulfur Filter, 393 nm
9	Diode Clamp Assembly
10	Screw, M4 x 6 mm long
11	Main Bracket
12	Chimney Back
13	Support Bracket
14	Chimney Top
15	Extension Spring
16	Screw, M4 x 45 mm long
17	Label Plate
18	Tube, 30 in. long
19	Cable Tie
20	Ferrule, Graphite
21	Brass Nut, 9/16 in.
22	Capillary Adapter

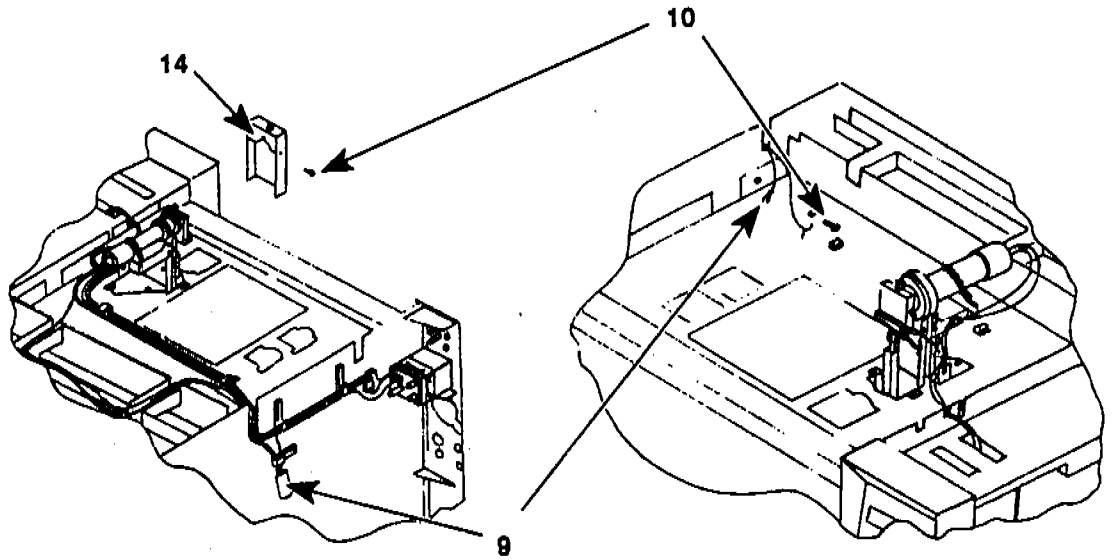


Figure 4

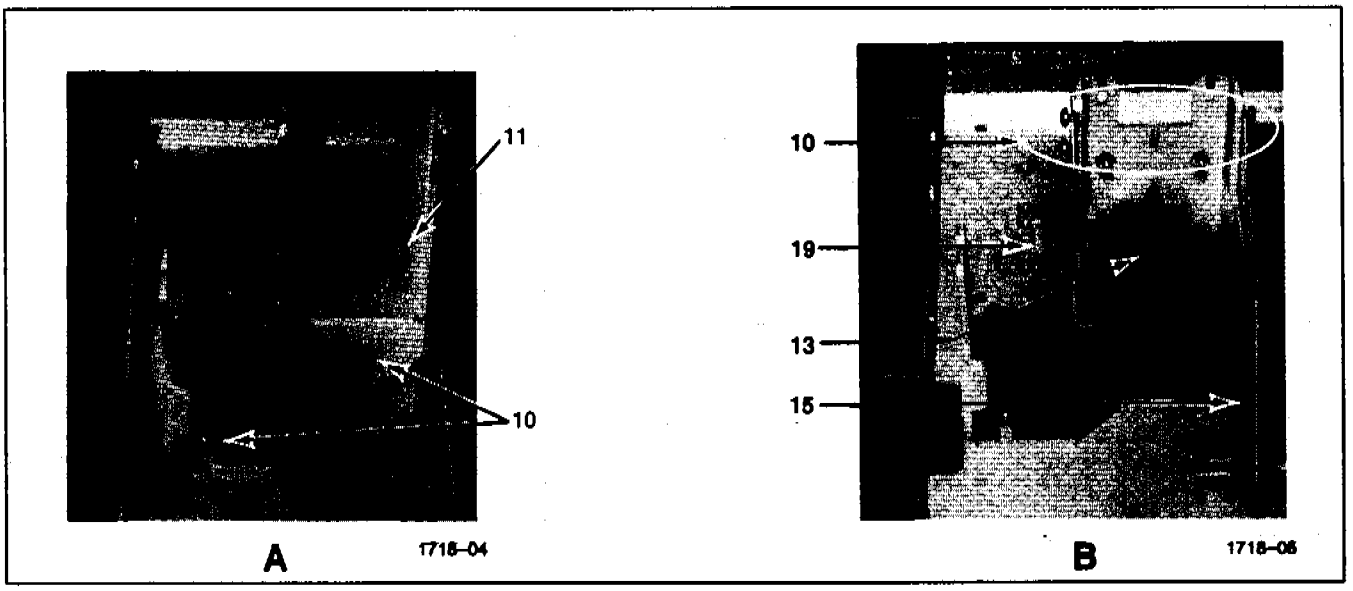


Figure 5

2. Loosely install the main bracket (11, figure 4) with two M4 X 6 mm screws (10) in the detector "B" location. Attach the PMT support bracket (13) to it with four M4 X 6 mm screws (10), press the metal cable tie (19) into its mounting hole and attach the extension spring (15).

3. Assemble the chimney back (12) to the main bracket (11) with four M4 X 6 mm screws (10). See Figure 4.

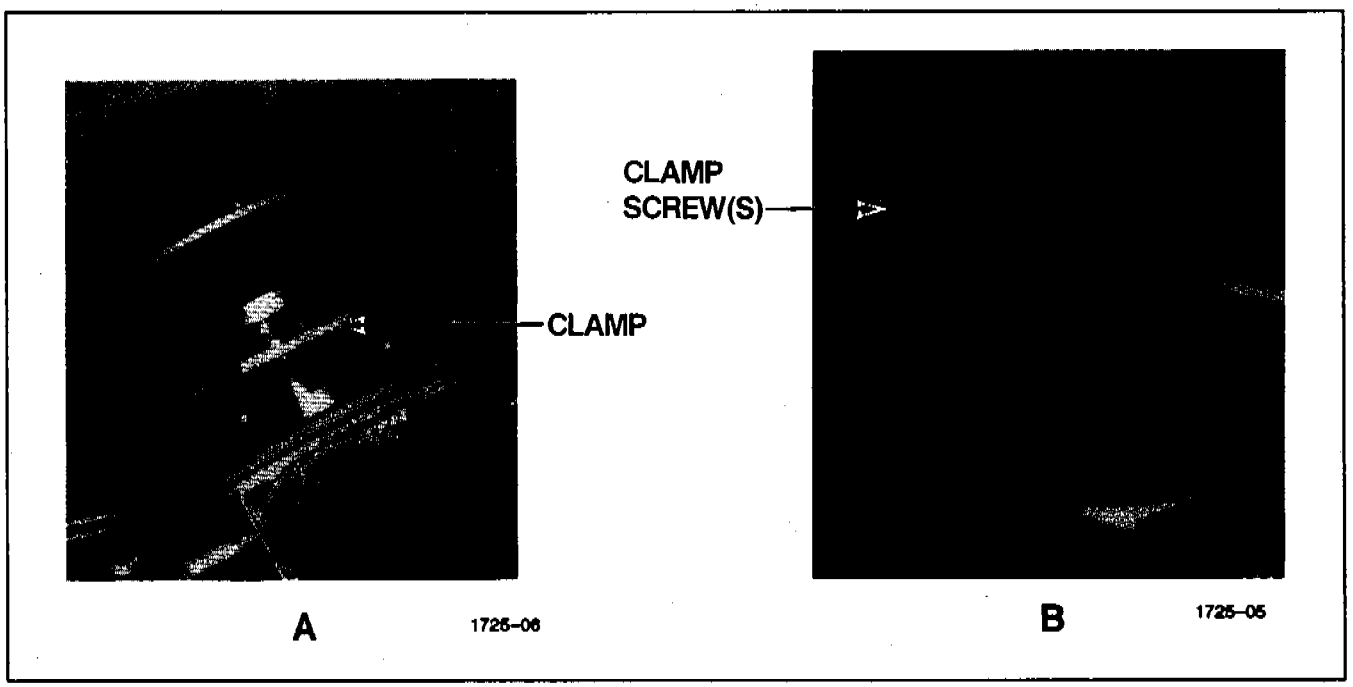


Figure 6

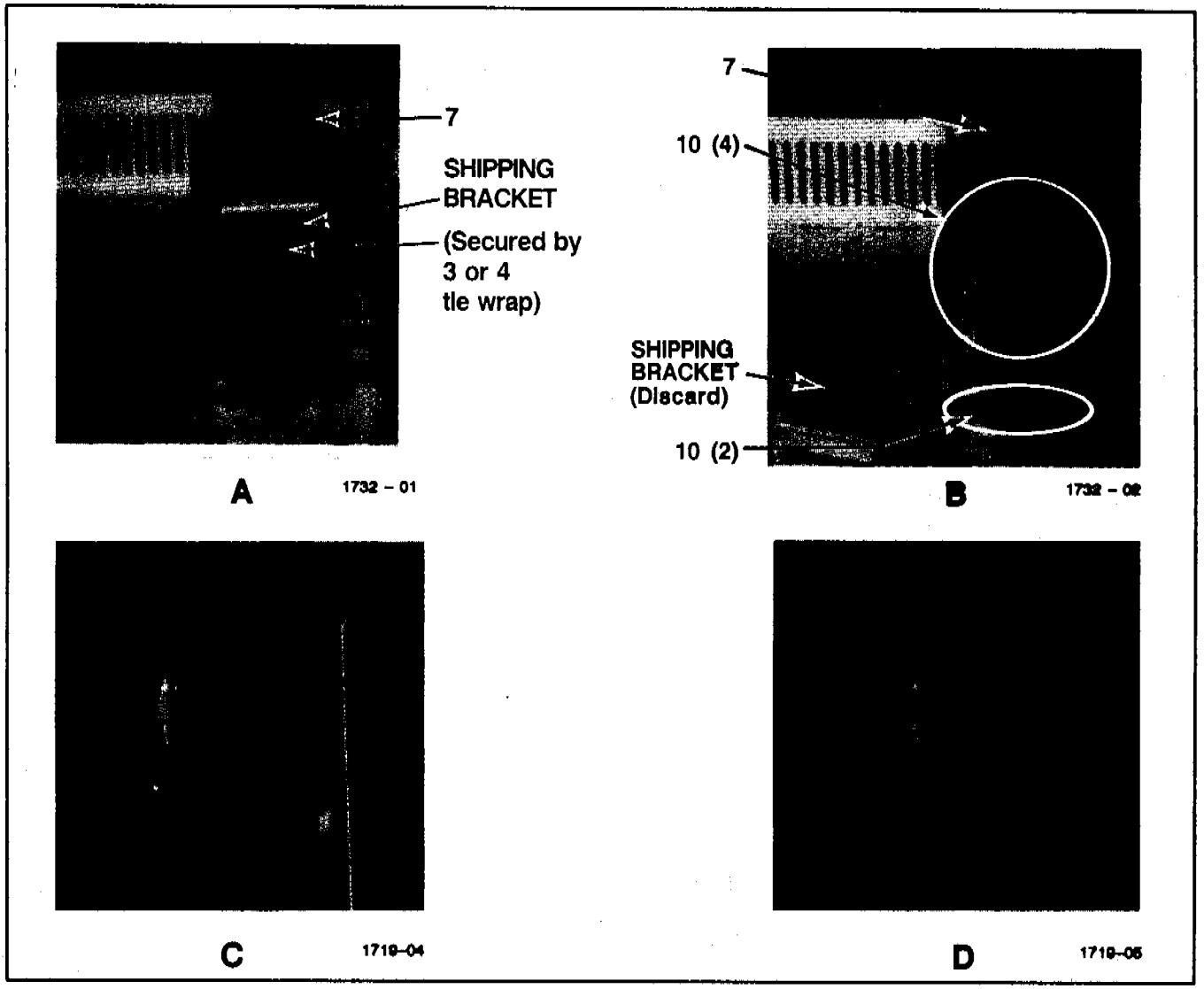


Figure 7

4. Loosen clamp (figure 6A) and place detector/flow assembly (22) (figure 6B) on the main/support bracket and secure. Fill spaces around the detector with the insulation removed in step 1. Tighten clamp (figure 6B) and remove shipping bracket (figures 7A and 7B).

5. Inside the oven remove the brass thread protector (figure 7C) and install the appropriate column fitting (capillary adapter shown, (22)) (figure 7D) with 1/4-inch brass tubing nut (21) and graphite ferrule (20).

6. Remove the blank filler panel from the detector "B" pneumatics location and, in its place, install the supplied FPD flow panel (11). The panels are self-adhesive and removal/installation is easily done without tools.

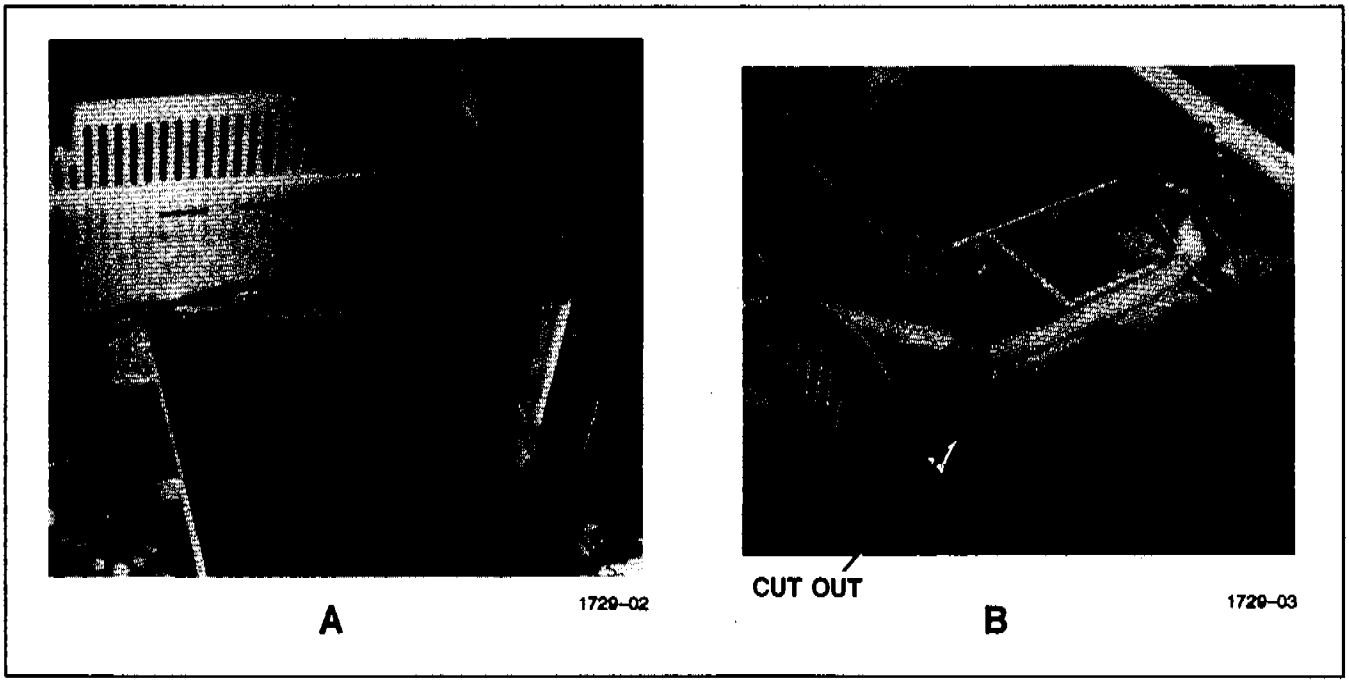


Figure 8

7. Route the flow manifold block tubing across the oven as show in figure 8, securing it in place with the metal clips that are already installed on the oven. Route the tubing through the cutout and leave extra tubing near the detector.

Figure 9

NOTE

Open the flow manifold block valves 1/4–turn before mounting the block and close the valves after the block is installed.

8. The flow manifold block is installed in the "B" position as shown in figures 9 and 10. A single M4 X 45 mm long screw (16) is used to secure the block to the flow panel. Tighten the screw to 2.7 Nm (24 in.– lbs.); or if torque screwdriver is not available, tighten the screw securely with a No.2 Pozidriv screwdriver.

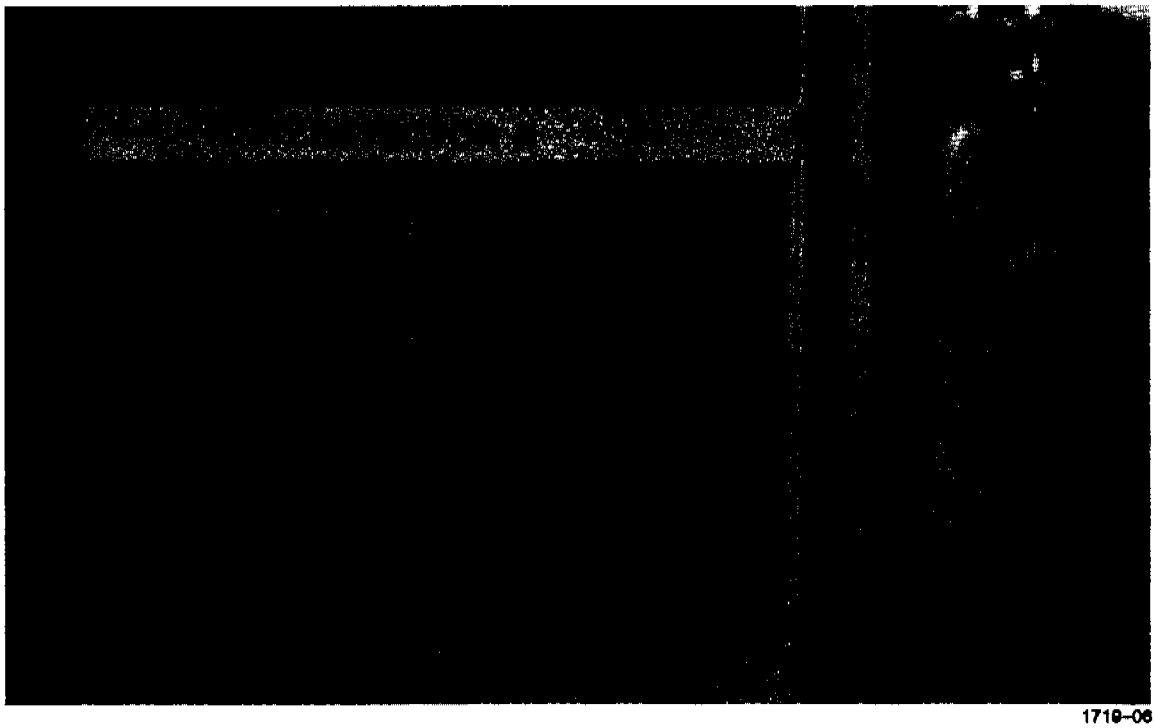


Figure 10

9. Connect FPD gas service to the flow manifold block. See figure 18 if gas will be routed through an Electronic Flow Sensor (EFS) and figure 32 for a flow diagram without EFS.

WARNING

HYDROGEN GAS MUST BE CONNECTED TO THE LOWER LEFT INLET FITTING ON AN FPD FLOW MANIFOLD BLOCK. THIS FITTING IS POSITIVELY IDENTIFIED BY VIEWING THE CONTROL PANEL MARKINGS – EACH INLET FITTING IS DIRECTLY BEHIND THE KNOB FOR THE GAS IT CONTROLS. ALSO, HYDROGEN LINES ARE MARKED WITH RED DYE FOR POSITIVE IDENTIFICATION.

NOTE

The "air" on/off valve may be labeled "oxygen" on the flow panel.

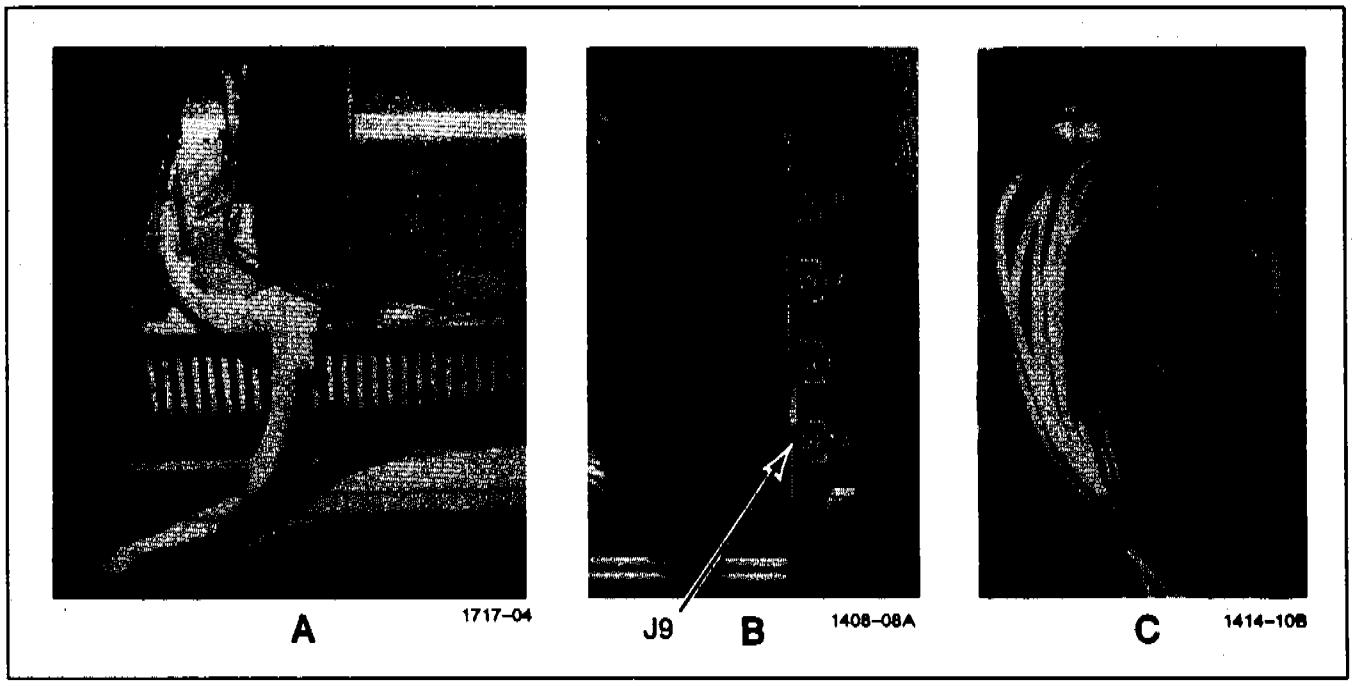


Figure 11

10. Heater/sensor leads are routed to the plastic cable tray at the back of the mainframe (figure 11A) and routed down the angled leg to the rear of the main board where they are connected to "J8" and "J9". Connect FPD heater and sensor leads to the instrument main circuit board as follows: sensor leads (white) to "J7" and heater leads (red) to "J9" (figure 11B).

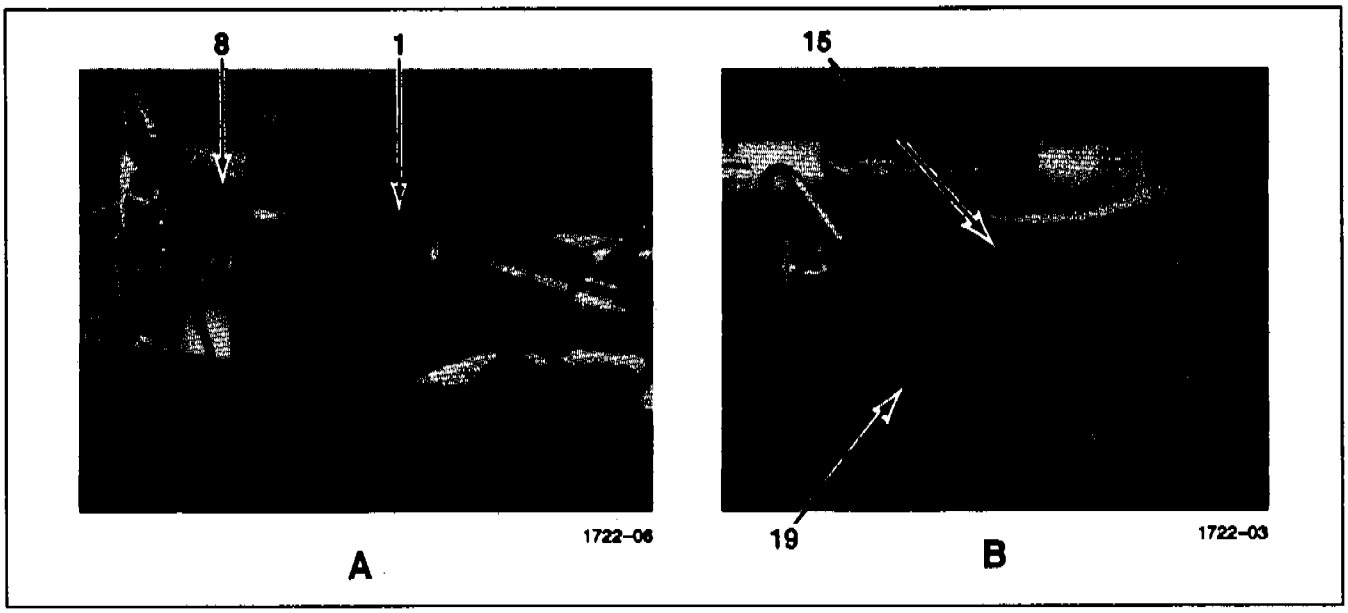


Figure 12

11. Install the appropriate optical filter (8) and the PMT assembly (1) as shown in figure 12. Install the extension spring (15) around the PMT assembly and route the high voltage and signal cables through the cable tie (19) as shown.

    Filter Orientation. When the filter has an arrow on it's edge it should be installed with the arrow pointing toward the PMT.

    If the filter has one side 'mirrored' (NO arrow), it should be installed with the mirror toward the flame (burner chamber).

    Filter Identification:

    "S" mode filters are labeled 393 (nM) or 3930 (A). (Part No. 19256-80000)

    "P" mode filters are labeled 525 (nM) or 5250 (A). (Part No. 19256-80010)

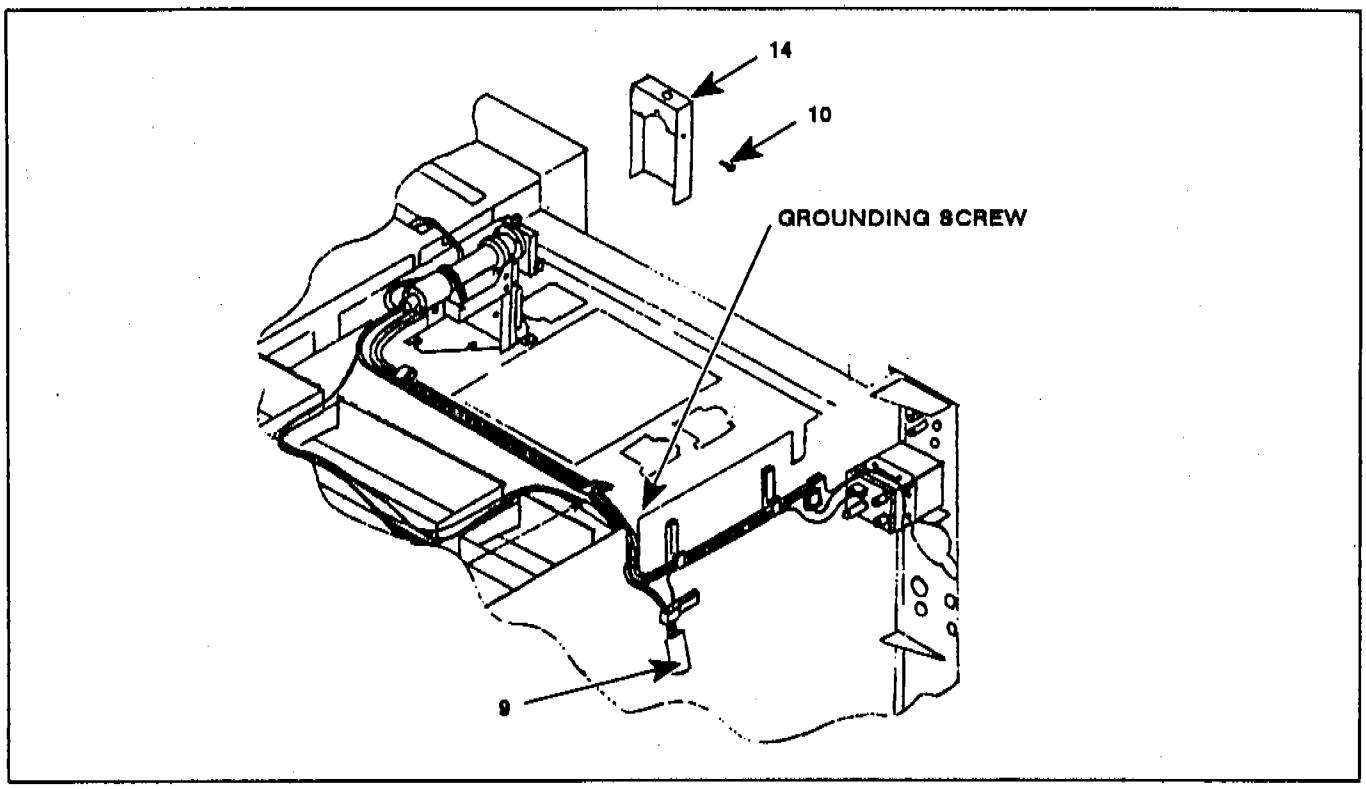


Figure 13

12. Install the diode clamp assembly (9) between the FPD burner chamber glow-plug and the pushbutton switch on the flow manifold block. Ground the harness with the spade lug lead and a screw to the mainframe as shown in figures 13 and 14.

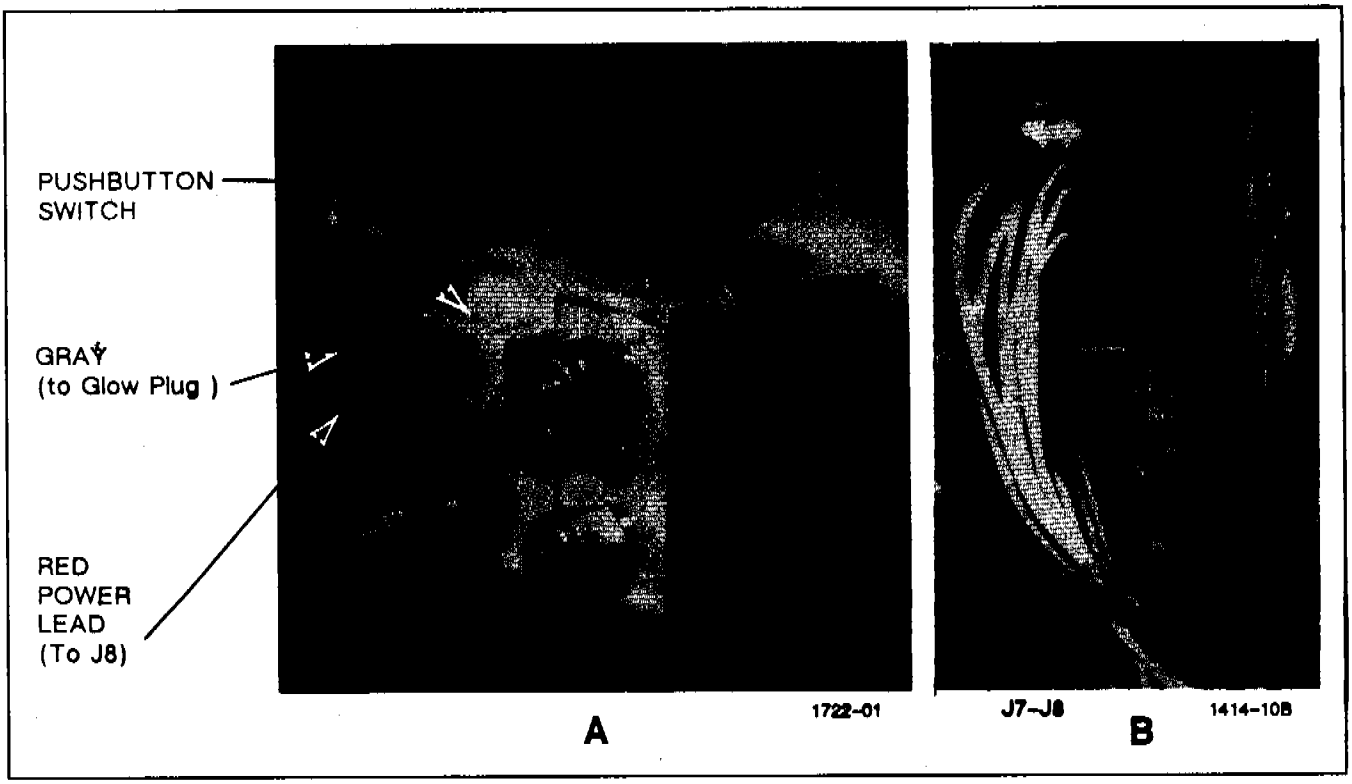


Figure 14

13. Connect the red power lead between the flow manifold switch (the second contact is for the glow plug) and main circuit board connector J8 terminal marked "FID IGN" (figure 14B).

14. During the preceeding installation procedures several screws were deliberately left loose to aid in installation/alignment of the various components. At this time, the screws can be securely tightened.

15. Install the detector board (in the "B" detector slot) part way and connect the FPD power and signal cables as shown in figure 15. Push the board in all-the-way.

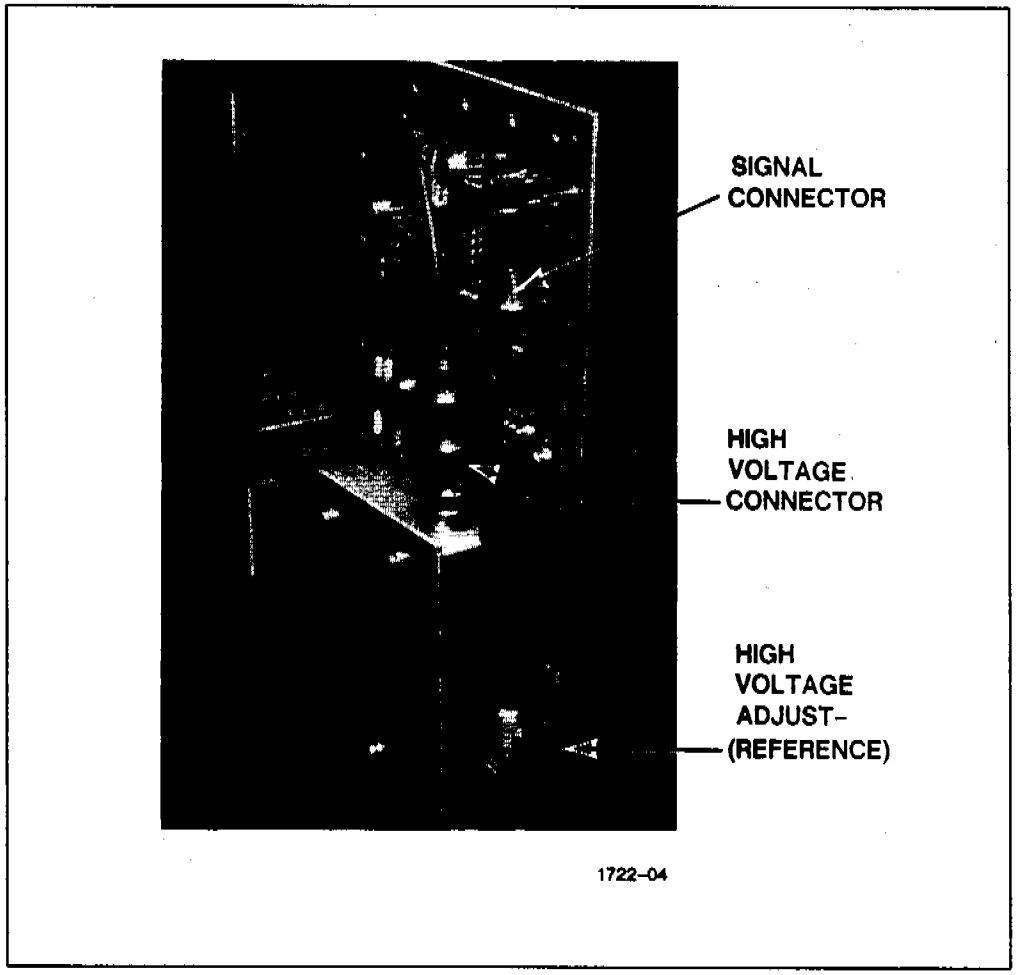


Figure 15

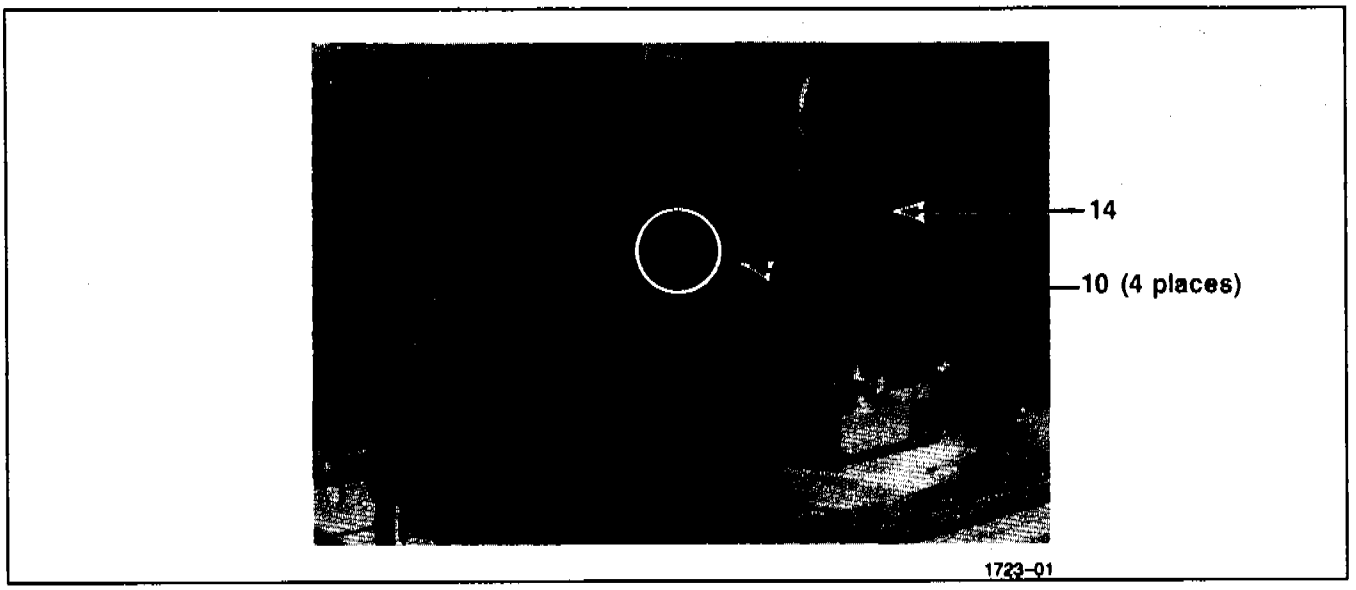


Figure 16

16. Install the chimney top (14), (figure 16) with four M4 X 6 mm screws (10), to complete the detector installation.

17. Install the new hinged top cover, with plastic injection port pan installed, to the HP 5890A with screws and washers removed from the HP 5890A (figure 17).

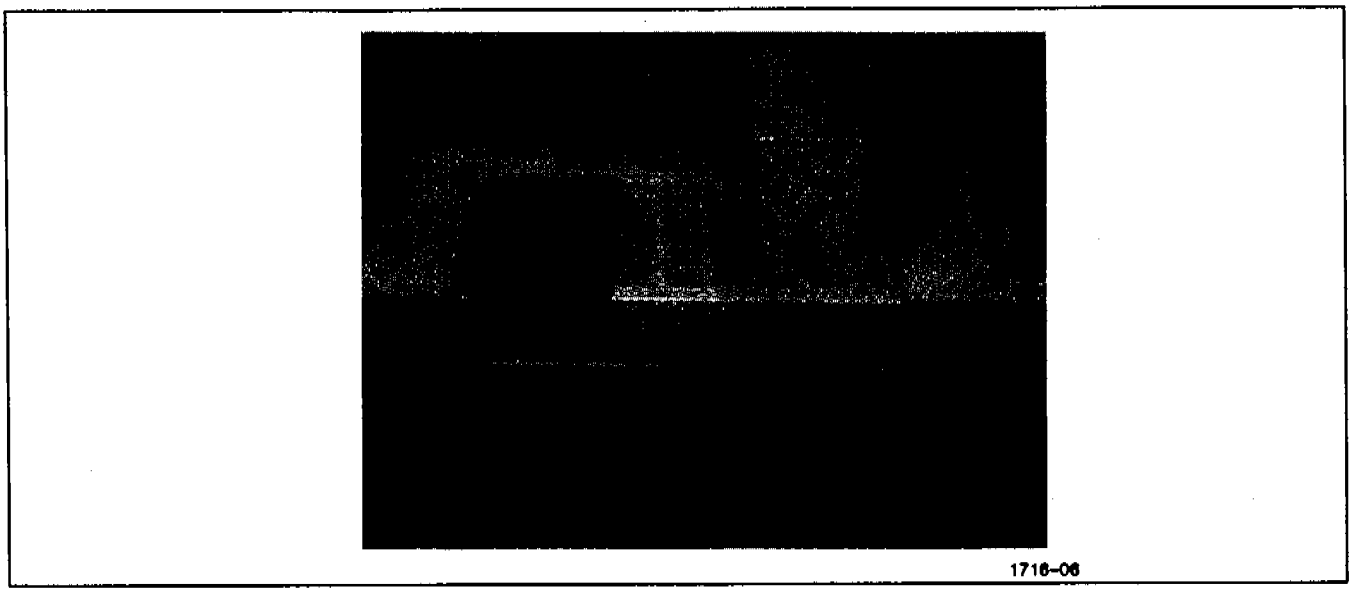


Figure 17

18. Attach the FPD Cover Assembly to the FPD hinged top cover. The cover is attached by a single thumb screw on the right side and a tab that is inserted under the sheetmetal on the left side as the cover is installed (when viewed from the front of the GC).

PLUMBING FPD GASES THROUGH THE ELECTRONIC FLOW SENSOR (EFS)

If plumbing the FPD hydrogen and air (or oxygen) flow lines through the Electronic Flow Sensor (EFS), do the following:

1. Disconnect the FPD hydrogen tube at the exit port of the FPD flow manifold block.

2. Connect the EFS channel A inlet tube to the flow manifold block hydrogen exit port.

3. Connect the FPD hydrogen tube to the EFS outlet tube.

4. Disconnect the FPD air (oxygen) exit fitting at the exit port of the flow manifold block.

5. Connect the EFS Channel B inlet tube to the flow manifold block air (oxygen) exit port.

6. Connect the FPD air (oxygen) tube to the EFS channel B outlet tube.

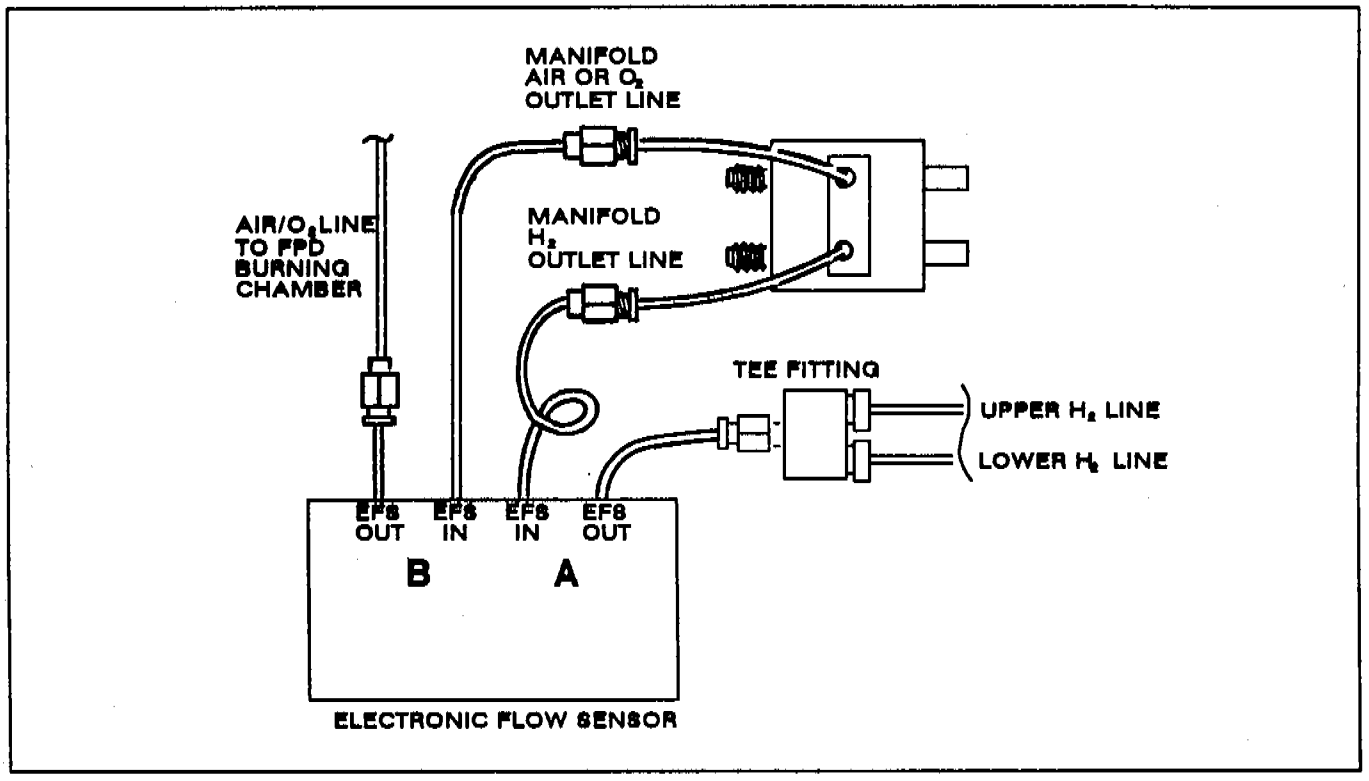


Figure 18. Connecting the FPD to a EFS

COLUMN INSTALLATION

INTRODUCTION

The HP 5890A provides flexibility in choices among inlets, columns, and detectors through use of liners and adapters allowing any standard column to be used without sacrificing performance. Additional flexibility is gained through positions of inlets and detectors relative to each other, and through the large internal volume of the oven. Refer to Section 4 of the HP 5890A GC Reference Manual (Volume 1) for the details of column installation for all detectors in general.

SPECIAL FPD ADAPTER WELDMENTS

The FPD uses special adapter weldments for use with capillary columns and 1/8-inch O.D. PTFE columns. The FPD Capillary Adapter Weldment, Part Number 19256-80570 allows easy installation of fused silica columns as large as 530u I.D. to be run right to the base of the FPD flame, minimizing sample tailing or loss of chemically active sites. The FPD 1/8-inch O.D. Adapter Weldment, Part Number 19256-80590 allows installation of PTFE columns concentrically around the FPD fused silica liner, eliminating exposed hot stainless steel.

Both of these adapter weldments are similar in appearance to FID adapters. See figure 19 for distinguishing features.

CAPILLARY COLUMN INSTALLATION

With the FPD capillary adapter weldment installed, fused silica columns as large as 530u I.D. may be installed up through the FPD Fused Silica Insert, Part No. 19256-60590 to the base of the flame as follows:

1. Install on the column a column nut (Part No. 18740-20870) and graphite ferrule (1.0 mm I.D., Part No. 5080-8773 or 0.5 mm I.D., Part No. 5080-8853).

2. Inserting the column through the nut and ferrule may contaminate the end of the column: prepare a fresh column end by cutting off a short piece of the column end. See the instructions given in "Preparing Capillary Columns" in Section 4 of the HP 5890A Reference Manual (Volume 1).

3. Position the column so it extends about 155 mm from the end of the ferrule and column nut (threaded end).

   This height may be optimized higher or lower, depending on sample type and detector flow rates. If the column is too high, it can be exposed to the detector flame. If the column is too low, the sample can be exposed to some hot stainless steel which can result in slight peak tailing.

   Mark the column at a point even with the bottom of the nut. (White typewriter correction fluid makes a good marking material). See figure 19 for details.

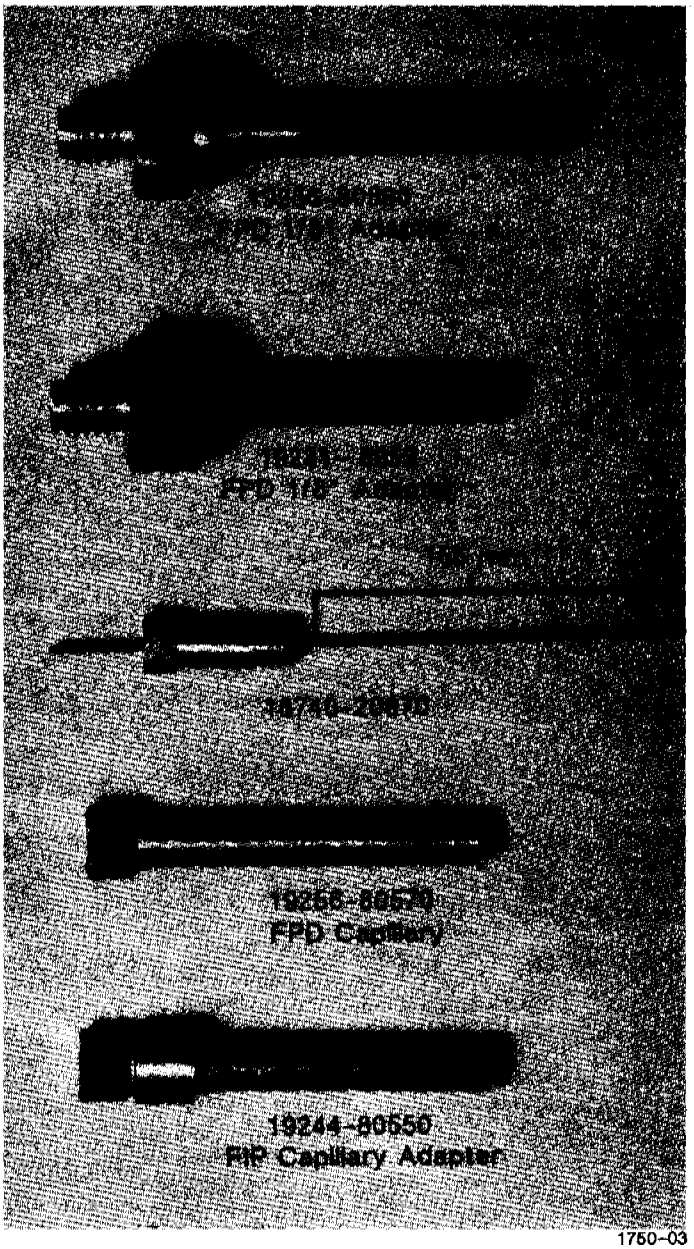


Figure 19. Column Adapters and Capillary Column Installation

PERFORMANCE VERIFICATION

CHECK-OUT PROCEDURES, GENERAL

WARNING

HYDROGEN GAS PRESENTS AN EXPLOSION HAZARD. OXYGEN IS A STRONG OXIDANT. FOLLOW ACCEPTED SAFETY PRACTICES IN HANDLING AND USING BOTH GASES.

NOTE

1.00 psi = 6.89 kPa = 0.0703 kg/cm2

CAUTION

THE UPPER TEMPERATURE LIMIT OF THE FPD IS 300 DEGREES C. THE HP 5890A TEMPERATURE CONTROL ALLOWS HEATING TO 400 DEGREES C. EXCEEDING THE 300 DEGREES C LIMIT OF THE FPD WILL DAMAGE THE DETECTOR SEALS.

CAUTION

DO NOT EXPOSE PMT TO AMBIENT LIGHT WITH VOLTAGE ON OR THE PMT WILL BE DESTROYED!

1. If not already done, restore power to the instrument.

2. Use a leak-detecting fluid to check for leaks at all installed fittings, particularly column fittings and supply fittings. The exhaust tube must be TEMPORARILY plugged while checking column fittings. (Refer to FPD LEAK TESTING procedure in the Troubleshooting section of this document.

3. Install the correct optical filter, depending on the choice of Sulfur or Phosphorus mode. For Sulfur Mode, use the 393 nanometer filter (Part No. 19256-80000). For Phosphorus Mode, use the 525 nanometer filter (Part No. 19256-80010).

4. Set the appropriate check-out flows for the selected flow mode:

   Air Flow Mode; AIR + HYDROGEN + CARRIER

   Oxygen Flow Mode; OXYGEN + HYDROGEN + AUXILIARY NITROGEN + CARRIER.

NOTE

Check-out flow rates are identical for sulfur or phosphorus modes of operation and the check-out procedure is the same for both AIR and OXYGEN flow modes of operation, other than flow settings. Flows may have to be optimized later for a particular analysis.

Recommended Check-out Flows
(Approximate Supply Pressures)

General Flow Rates	OXYGEN FLOW MODE Flow Supply Pressure ml/min	kPa	(PSIG)	AIR FLOW MODE Flow Supply Pressure ml/min	kPa	(PSIG)
OXYGEN	20	130	(19)	—	—	
HYDROGEN	75	185	(27)	75	185	(27)
AIR	--	--		100	385	(56)
CARRIER + AUXILIARY*	120-140	435	(63)	15-40	435	(63)

* Adjust auxiliary (nitrogen) gas with needle valve adjustment (turn valve clockwise to REDUCE flow).

SETTING FLOW RATES

To set FPD flows, begin with all manifold block flows off (valves fully clockwise) and with carrier flow off (mass flow controller knob fully clockwise). Measure each flow separately with a bubble meter attached to the FPD vent tube. For instruments with the FPD hydrogen and air (or oxygen) plumbed through the electronic flow sensor (EFS), once the absolute flows are measured with a bubble meter they may be monitored for changes with the EFS.

Set, and then turn off, each of the detector gas flows at the flow manifold block. Set the carrier gas flow last, since it cannot be turned off without changing the flow settings, then proceed.

In the oxygen mode, the auxiliary nitrogen gas is usually controlled from the same supply pressure regulator as the nitrogen carrier gas. To avoid disturbing the carrier flow rate, (always) first preset the supply pressure regulator to give the desired auxiliary nitrogen (with the auxiliary gas needle valve wide open –fully counterclockwise) then adjust the injection port mass flow controller to get the desired column flow rate or split ratio.

NOTE

Changing the supply pressure to mass flow controller will change the gas quantity delivered.

In the air mode, when carrier flow is very low and some make-up gas is desired, use the auxiliary gas needle valve to set the desired auxiliary gas flow rate.

After setting detector flows, shut all detector manifold block flows off (valves fully clockwise) until ready to light the flame.

IGNITING THE FPD FLAME

NOTE

The detector module is most easily lit if heated to at least 200 degrees C.

If flows are set correctly, the FPD flame is relatively easy to ignite, BUT the sequence used is important to avoid a loud "POP" on ignition.

The correct ignition sequence, beginning with all FPD flows OFF, (except column flow) is:

1. If required, open auxiliary NITROGEN valve.
2. Open AIR or (OXYGEN) valve.
3. Press in and hold ignitor button.
4. Open HYDROGEN valve.
5. Release ignitor button.

Proper ignition should result in a slightly audible "pop". Flame ignition can be verified by holding a mirror or shiny surface near the exhaust tube and observing condensation. Ignition also usually results in a small increase in signal offset on the LED display.

NOTE

Always open the HYDROGEN valve AFTER opening AIR (OXYGEN) and pressing the ignitor. Failure to do this will result in a loud audible "POP". This should not damage the detector but is unpleasant to hear.

CAUTION

DO NOT ATTEMPT IGNITING THE FPD FLAME BY APPLYING A FLAME AT ITS EXHAUST TUBE. A DANGEROUS EXPLOSION CAN RESULT.

SULFUR AND PHOSPHORUS MODE CHECK-OUT PROCEDURE

1. Set the detector heated zone to 200 degrees C and the oven to 110 degrees C. Before lighting the FPD flame, observe the "flame off" detector signal offset. It should be between 140 and 260 counts on the HP 5890A LED display.

2. With the flows set and the FPD lit, the "flame on" detector signal offset should increase no more than 100 counts over the "flame off" offset. If it does, bake out the system for at least one hour at:

   Injector Temperature..... 200 degrees C

   Detector Temperature.....250 degrees C

   Oven Temperature.....225 degrees C

3. After baking out, lower the oven temperature to 110 degrees C and the detector temperature to 200 degrees C.

4. Proceed with check-out sample injection, using the test conditions appropriate for the inlet type being used.

HP 5890 OPERATING CONDITIONS

Detector Type FPD
Temp. 200 DEGREES C
Inlet Type PACKED OR PURGED PACKED
Temp. 200 DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp. Programmed (1 ramp)
Init. Temp. 110 DEGREES C
Init Time 0 min
Ramp Rate 10
Fin. Temp. 170
Fin. Time. 3
Range 5
COLUMN:
Part No. 19095S-100
Dimensions 5 M Length, 530µ ID
Sta. Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/- 1	ml/min
Hydrogen	75 +/- 2	ml/min
Air	100 +/- 3	ml/min
Make-Up (N2)	0	ml/min
Split Vent	N/A	ml/min
Septum Purge	1-2	ml/min

SAMPLE:
Type FPD Sample
Inj. Volume 1ul
Part No. 19395-60580
COMPOSITION: 20 ng/uL
(20.0 ppm W/V) each of
1-dodecanethiol and
tributylphosphate in iso-octane
CHROMATOGRAM/REPORT FEATURES:
COMPOUND MINIMUM AREA COUNT

	S filter	P filter
1-dodec-	2,000,000	
tributyl	--------	6,300,000

BASELINE NOISE MAXIMUM

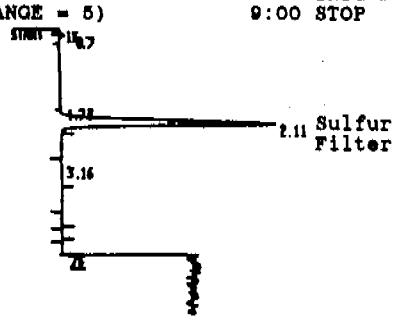
	S filter	P filter
Analog	10 mm	12 mm
Digital	15 mm	18 mm

peak #1 = iso-octane (solvent)
peak #2 = 1-dodecanethiol
peak #3 = tributylphosphate

TYPICAL PERFORMANCE, 3392A Analog

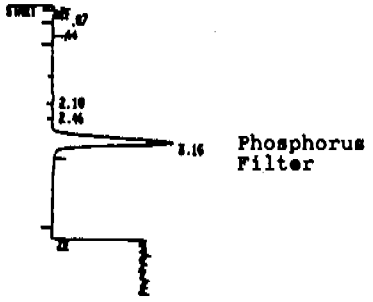
Time Table

ZERO = 10, 0.05	LIST:TIME @
ATT 2^ = 9	0:00 INTG # = 4
CHT SP = 1.0	7:00 PK WD = 0.04
PK WD = 0.16	7:00 ATT 2^ = 0
THRSH = 2	7:00 ZERO = 50
AR REJ = 0	7:00 INTG # = 9
(RANGE = 5)	9:00 STOP



	RT	AREA	TYPE	AR/HT	AREA%
1)	0.07	50776	0 BP	0.033	0.415
	1.73	94871	PP	2.967	0.776
2)	2.11	1.2066E+07	PB	0.140	98.683
3)	3.16	21210	BB	0.188	0.173

TOTAL AREA = 1.2232E=07
MUL FACTOR = 1.0000E+00



	RT	AREA	TYPE	AR/HT	AREA%
1)	0.07	526640 D	BB	0.060	2.398
	0.44	62010	PB	0.109	0.283
2)	2.10	132220	PV	0.136	0.603
	2.46	59930	VP	0.171	0.277
3)	3.16	2.1142E+07	PB	0.214	96.4

TOTAL AREA = 2.1922E+07
MUL FACTOR = 1.0000E+00

Figure 20. FPD Checkout Conditions with a Packed (or Purged/Packed) Column Inlet

HP 5890 OPERATING CONDITIONS

Detector Type FPD
Temp. 200 DEGREES C
Inlet Type SPLIT ONLY OR
SPLIT/SPLITLESS
Temp. 200 DEGREES C
Operating Mode SPLIT (PURGE ON)
Purge Time On 0 min
Purge Time Off 0 min
Oven Temp. Programmed (1 ramp)
Init. Temp. 110 DEGREES C
Init Time 0 min
Ramp Rate 10
Fin. Temp. 170
Fin. Time. 3
Range 5
COLUMN:
Part No. 19095Z-121
Dimensions 10 M Length, 530μ ID
Sta. Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/- 1	ml/min
Hydrogen	75 +/- 2	ml/min
Air	100 +/- 3	ml/min
Make-Up (N2)	0	ml/min
Split Vent	200 +/- 20	ml/min
Septum Purge	5 +/- 1	ml/min

SAMPLE:
Type FPD Sample
Inj. Volume 2μl
Part No. 19305-60580
COMPOSITION: 20 ng/μl
(20.0 ppm W/V) each of
1-dodecanethiol and
tributylphosphate in iso-octane
CHROMATOGRAM/REPORT FEATURES:
COMPOUND MINIMUM AREA COUNT

	S filter	P filter
1-dodec-	38,000(min)	--------
tributyl	-------	1,150,000

BASELINE NOISE MAXIMUM

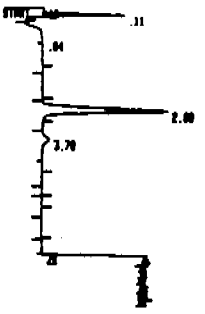
	S filter	P filter
Analog	10 mm	12 mm
Digital	15 mm	18 mm

peak #1 = iso-octane (solvent)
peak #2 = 1-dodecanethiol
peak #3 = tributylphosphate

TYPICAL PERFORMANCE, 3392A Analog
Time Table

ZERO = 10, 0.05
ATT 2^ = 5
CHT SP = 1.0
PK WD = 0.16
THRSH = 2
AR REJ = 0
(RANGE = 5)

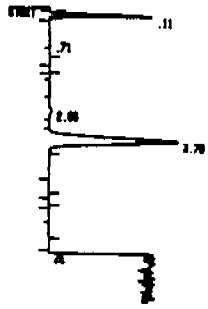
LIST:TIME @
0:00 INTG # = 4
7:00 PK WD = 0.04
7:00 ATT 2^ = 0
7:00 ZERO = 50
7:00 INTG # = 9



Sulfur Filter

AREA% RT		AREA	TYPE	AR/HT	AREA%
1)	0.11	142610 D	BB	0.055	16.027
	0.84	149460	PV	0.505	16.797
2)	2.88	554190	PB	0.147	62.282
3)	3.70	43547	BP	0.210	4.894

TOTAL AREA = 889800
MUL FACTOR = 1.0000E+00



Phosphorus Filter

AREA% RT		AREA	TYPE	AR/HT	AREA%
1)	0.11	749180 D	BB	0.061	19.519
	0.71	34857	PV	0.535	0.908
2)	2.88	43072	PV	0.164	1.122
3)	3.70	3011200	VB	0.196	78.451

TOTAL AREA = 2.1922E+07
MUL FACTOR = 1.0000E+00

Figure 21. FPD Checkout Conditions with Split Injection

HP 5890 OPERATING CONDITIONS

Detector Type FPD
Temp. 200 DEGREES C
Inlet Type DED. ON-COL. CAP.

Temp. N/A DEGREES C
Operating Mode N/A
Purge Time On N/A min
Purge Time Off N/A min
Oven Temp. Programmed (1 ramp)
Init. Temp. 90 DEGREES C
Init Time 1.0 min

Ramp	(1)	(2)
Rate	20	10
Fin. Temp.	110	170
Fin. Time.	0	3

Range 5
COLUMN:
Part No. 190958-100
Dimensions 5 M Length, 530μ ID
Sta. Phase Methyl Silicone

FLOW RATES (FPD AIR MODE FLOWS)

Carrier (N2)	20 +/- 1	ml/min
Hydrogen	75 +/- 2	ml/min
Air	100 +/- 3	ml/min
Make-Up (N2)	0	ml/min
Split Vent	N/A	ml/min
Septum Purge	5 +/- 1	ml/min

SAMPLE:
Type FPD Sample
Inj. Volume 1ul
Part No. 19305-60580
COMPOSITION: 20 ng/uL
(20.0 ppm W/V) each of
1-dodecanethiol and
tributylphosphate in iso-octane
CHROMATOGRAM/REPORT FEATURES:
COMPOUND MINIMUM AREA COUNT

	S filter	P filter
1-dodec-	2,000,000(min)	
tributyl	--------	6,300,000

BASELINE NOISE MAXIMUM

	S filter	P filter
Analog	10 mm	12 mm
Digital	15 mm	18 mm

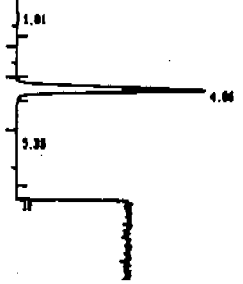
peak #1 = iso-octane (solvent)
peak #2 = 1-dodecanethiol
peak #3 = tributylphosphate

TYPICAL PERFORMANCE, 3392A Analog

Time Table

ZERO = 10, 0.05
ATT 2^ = 9
CHT SP = 1.0
PK WD = 0.16
THRSH = 2
AR REJ = 0
(RANGE = 5)

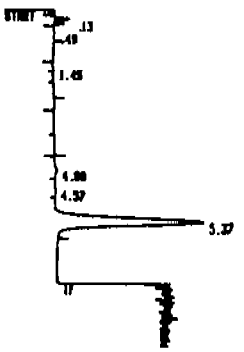
LIST:TIME ●
0:00 INTG # = 4
7:00 PK WD = 0.04
7:00 ATT 2^ = 0
7:00 ZERO = 50
7:00 INTG # = 9
9:00 STOP



Sulfur Filter

	RT	AREA	TYPE	AR/HT	AREA%
1)	0.13	179500	BB	0.066	2.686
	1.91	39086	PV	1.980	0.585
2)	4.06	6441500	PB	0.155	96.393
3)	5.35	22487	PB	0.229	0.337

TOTAL AREA = 6682500
MUL FACTOR = 1.0000E+00



Phosphorus Filter

	RT	AREA	TYPE	AR/HT	AREA%
1)	0.13	578300	BB	0.063	3.007
	0.49	104640	PB	0.113	0.358
	1.45	6367	BV	0.156	0.022
2)	4.08	284220	PP	0.153	0.973
	4.57	101040	PP	0.194	0.346
3)	5.37	2.7834E+07	PB	0.218	95.294

Figure 22. FPD Checkout Conditions with a Dedicated On-column Inlet

OPTIMIZATION

OPTIMIZING FPD SENSITIVITY AND SELECTIVITY

FPD sensitivity and selectivity are affected by several important parameters. These are listed below, with suggested ways to optimize for each application.

A. FPD Flow Rates. FPD flow rates are the most critical for optimizing either sensitivity or selectivity (these do not necessarily have the same optimal conditions). The most critical flow parameter is the hydrogen/air (or hydrogen/oxygen) ratio. Less critical are the auxiliary nitrogen/carrier combined flows.

A suggested way to set near-optimal flows is to begin with recommended checkout flow rates. Then vary each gas until a local maxima is reached. Optimize hydrogen first, then air (or oxygen), and last the auxiliary nitrogen flows. This may require a couple alterations. Adjusting these flows is most easily done by just varying the supply pressure regulator setting. Figure 23 plots approximate FPD flows versus supply pressure for all FPD gases.

NOTE

Take care that adjusting the supply gas pressure does not change some other flow supplied by the same regulator.

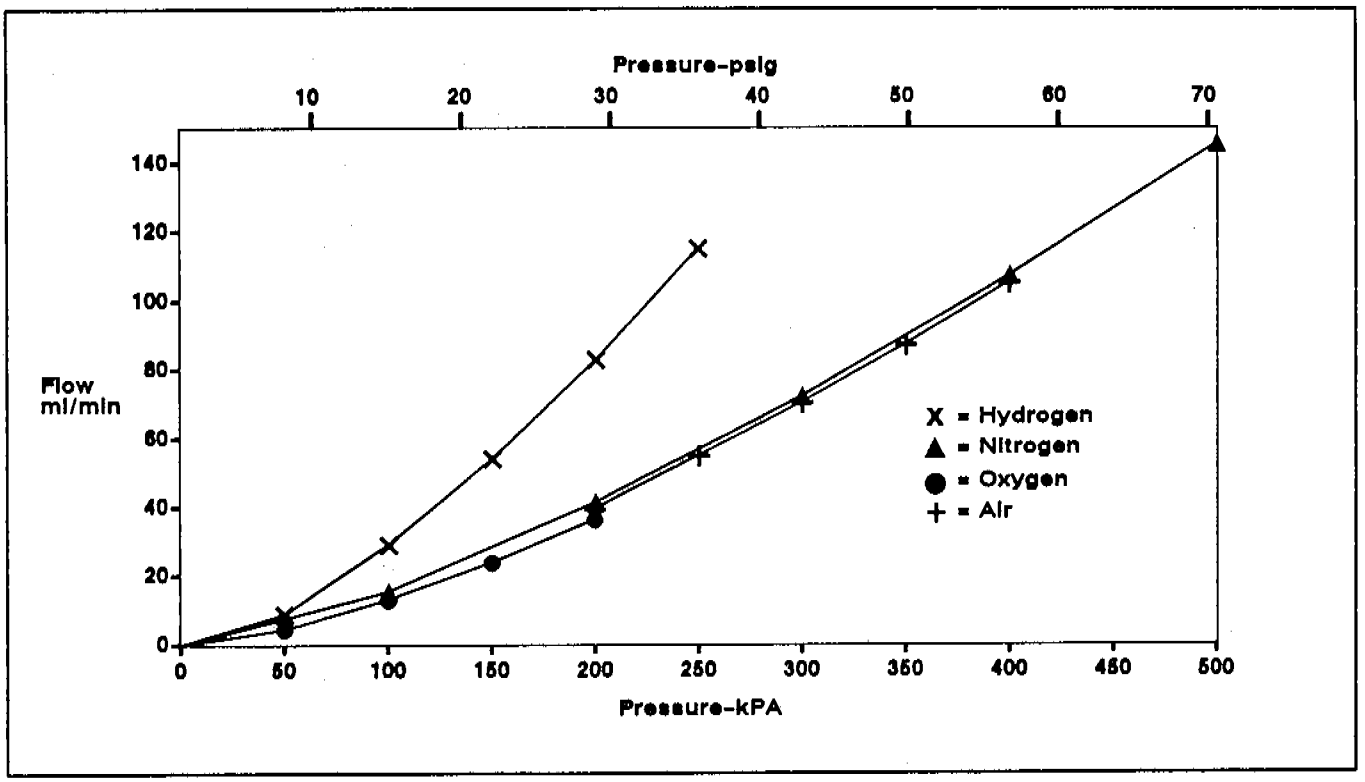


Figure 23. FPD Flows VS Supply Pressures

B. Detector Temperature. Detector heated zone temperature can have a significant effect on sensitivity. If analyzing thermally labile or very unstable compounds, a lower heated zone temperature may give the best results. If analyzing compounds with high boiling points, the detector temperature should be set at least 25 degrees C above the final oven temperature (but not greater than 300 degrees C!).

C. Quenching. Quenching can result to some degree if sulfur or phosphorus peaks coelute with large hydrocarbon peaks. For many applications, this affect can be reduced by better chromatographic separation of peaks by varying the oven temperature program conditions and/or column selection. In cases where there is a large continuous hydrocarbon background, better selectivity of sulfur to carbon may be obtained by adjusting the hydrogen or air (oxygen) flow rate.

TROUBLESHOOTING

FLAME IGNITION PROBLEMS

Two common flame ignition problems are:

A loud "pop" results on ignition and the flame will not light or stay lit.

If a loud "pop" occurs on ignition, it is usually caused by an incorrect ignition sequence. The correct ignition sequence is:

1. Open the auxiliary NITROGEN valve if required.
2. Open the AIR/OXYGEN valve fully counterclockwise (CCW).
3. Press in and hold the ignitor button.
4. Open the HYDROGEN valve fully counterclockwise (CCW). On doing this, there should be a slight "pop".

NOTE

A loud "pop" is caused by opening the hydrogen valve BEFORE pressing the ignitor, thereby igniting a volume rich in hydrogen. This should not damage the detector but is unpleasant to hear. Always open the hydrogen valve AFTER pressing the ignitor.

If the FPD flame won't light or stay lit, check/do the following:

1. Be sure there is a problem. Under some conditions, the flame lights so quietly that the "pop" is nearly inaudible. Also, especially in oxygen mode, the increase in signal offset on the LED display is usually only a few counts. Flame ignition is best verified by holding a mirror or shiny surface near the aluminum exhaust tube (with the rubber drip tube removed) and observing condensation (if the flame is lit).

2. If the flame doesn't light at all, be sure the glow plug circuit is working. This is most easily done by observing the LED display, which should go to greater than 65500 counts when the ignitor switch is pressed. If the LED display doesn't change with the ignitor switch pressed either the circuit is open (check the pin connections at the detector flow manifold block, the lead connection on the glow plug and the appropriate 5A fuse on the instrument's main circuit board. If the glow plug has failed replace with part number 0854- 0141.

3. Under some operating conditions, it is important to continue to hold the ignitor switch in for several seconds after opening the hydrogen valve fully counterclockwise.

4. Under some operating conditions, the flame may be more easily lit with the rubber drip tube removed. After lighting the flame, reinstall the drip tube and FPD cover assembly.

5. Under some conditions, the flame may be more easily lit with the detector temperature raised to 200 degrees C or higher (but do not exceed the detector's maximum temperature of 300 degrees C) to ignite the flame. After the flame is lit, set the detector to the required temperature.

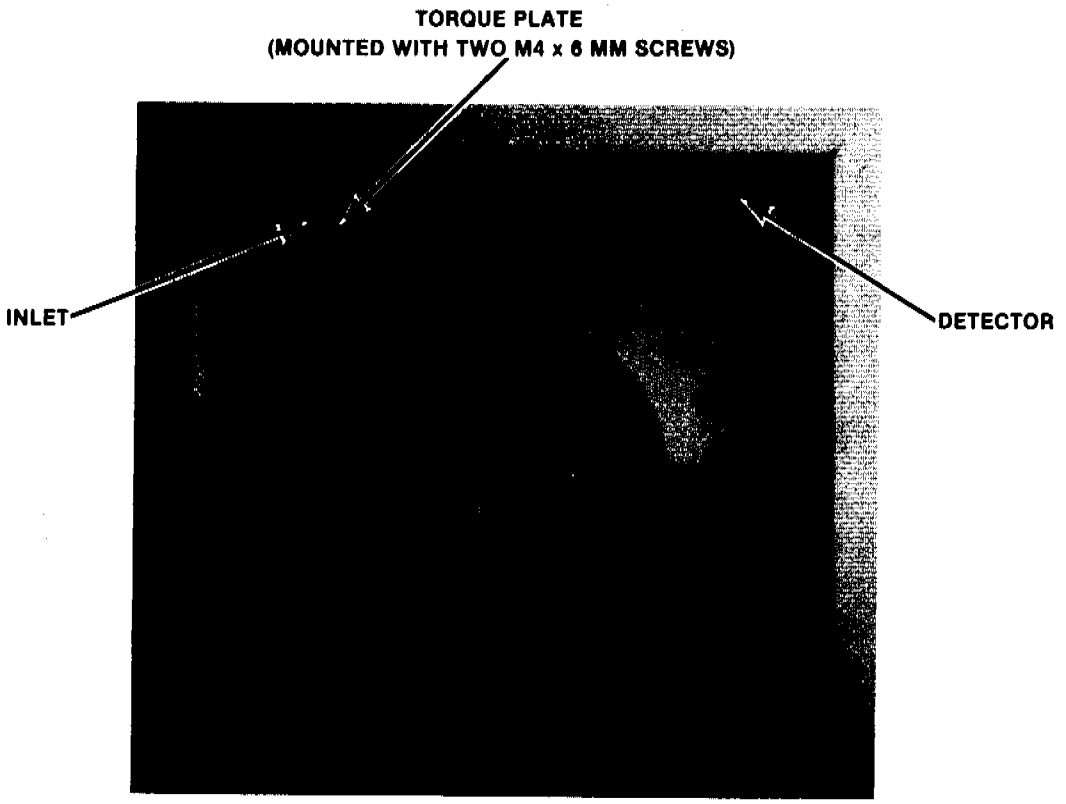
6. If none of the above are sufficient to light the flame, try increasing the hydrogen supply pressure by 20–40%. After igniting the flame, reduce the hydrogen supply pressure to the initial value.

7. If the flame still won't light after trying the preceeding steps, there may be a large leak in the system. This can result in measured flow rates being significantly different from actual flow rates, resulting in non-ideal ignition conditions. Thoroughly leak check the whole system.

FPD LEAK TESTING/GC HAS ELECTRONIC FLOW SENSOR

If the system has an electronic flow sensor (EFS) with any FPD gas plumbed through it (air, oxygen, hydrogen or carrier), the system can be easily and quickly be checked. First, close all supply gases except for the one plumbed through the EFS. Then cap off the detector exhaust tube with a 1/4-inch Swagelok plug (Part No. 0100-0196) and a 40% graphitized Vespel ferrule (Part No. 0100-1061).

With the flow system deadheaded and one pressurized gas plumbed through the EFS, the flow reading should drop very close to zero. If not, this indicates a leak in the system. Begin checking possible leak sources and monitor the EFS to determine when the leak has been eliminated. See caution on next page for maximum pressure.

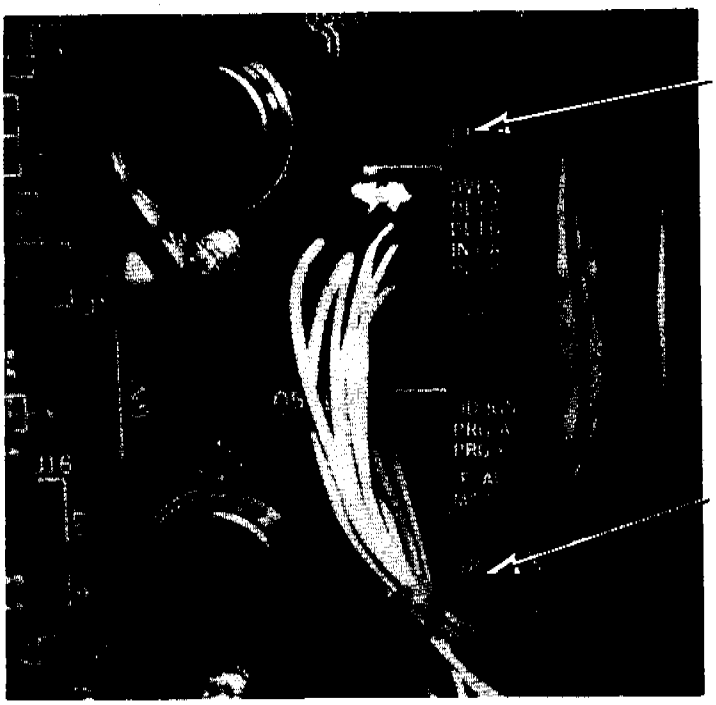


NOTE

FOR COMPLETE INSTRUCTIONS ON HOW TO INSTALL THE VARIOUS COLUMNS AVAILABLE FOR THE HP 5890A, REFER TO SECTION 4, COLUMN INSTALLATION IN THE HP 5890A SHELF REFERENCE MANUAL.

Figure 8. Detector/Inlet Fittings in Column Oven

J7/8



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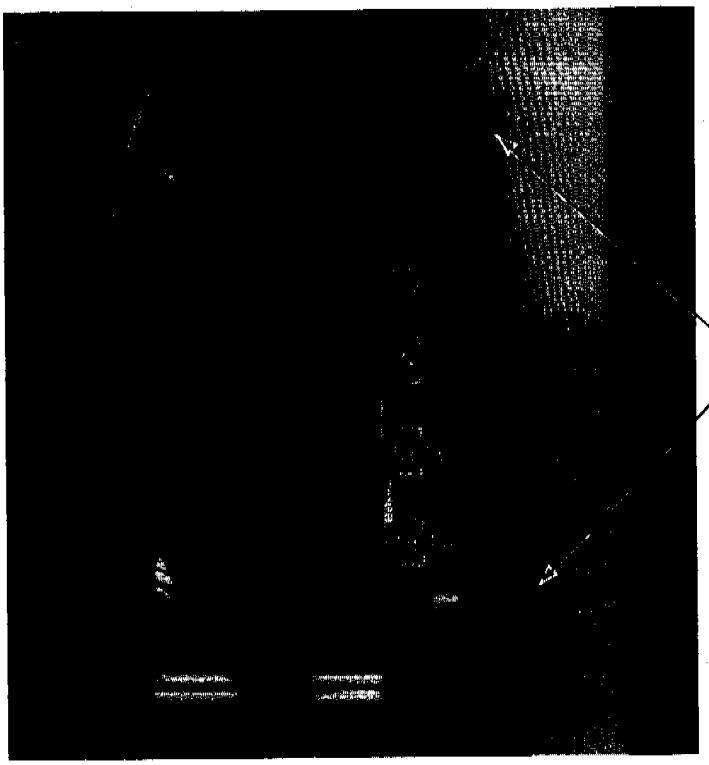
J9



SEE FIGURE 4 FOR LOCATION ON MOTHERBOARD.

1408-08

Figure 7. Accessory Connections on Main Circuit Board

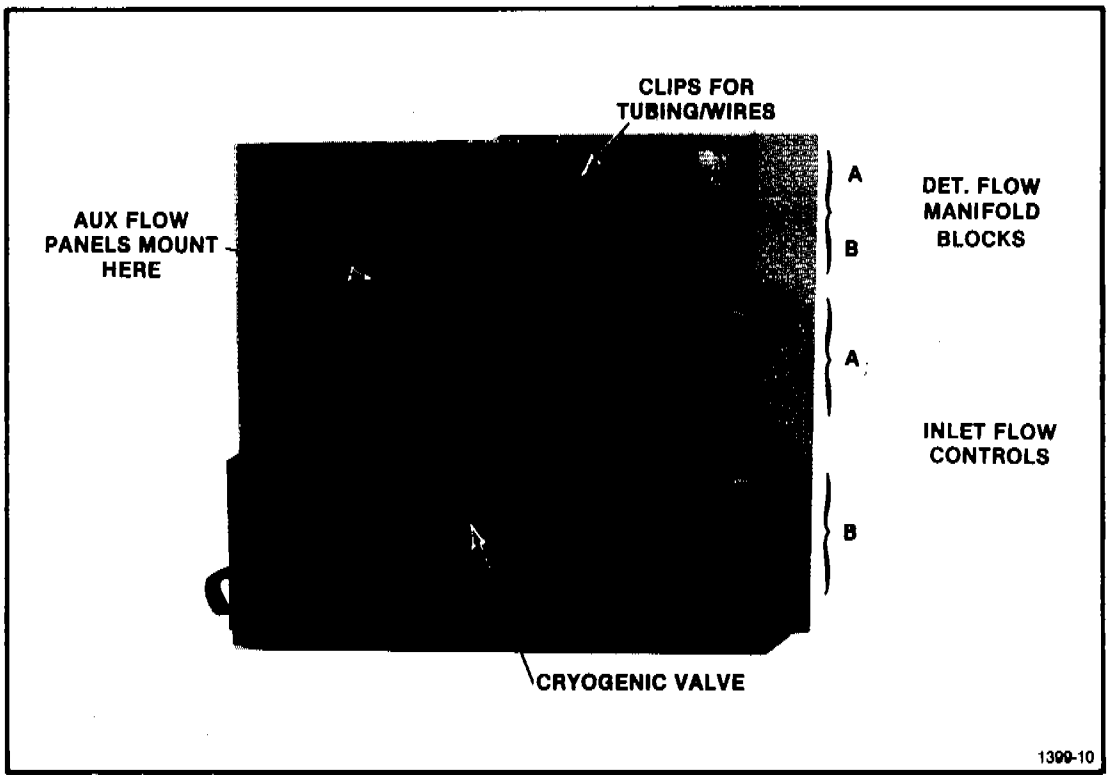


Figure 5. Left Side of Instrument

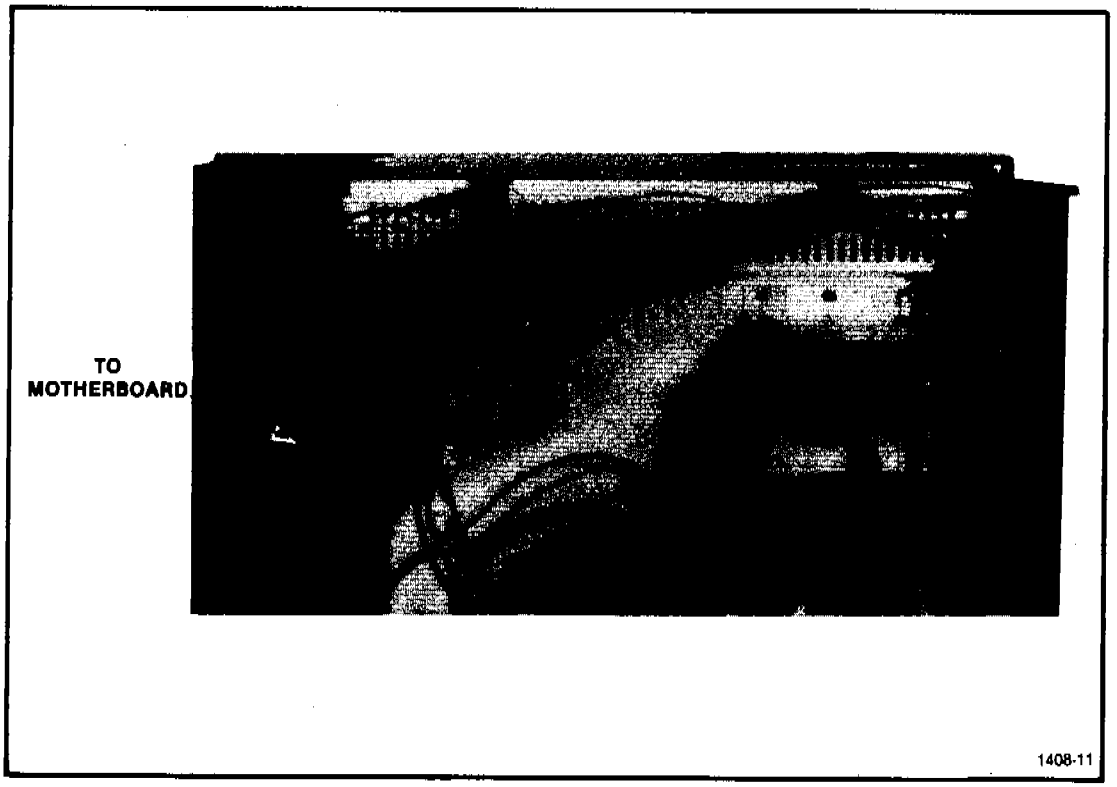


Figure 6. Back of Instrument

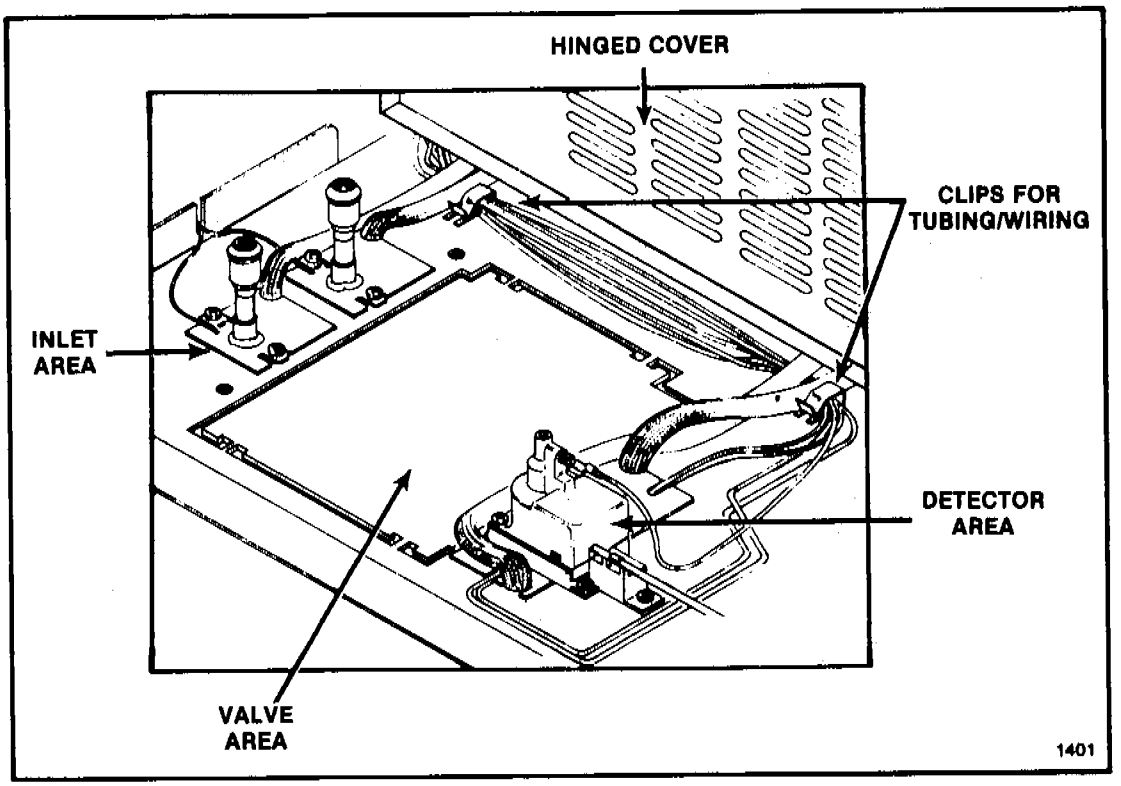


Figure 3. Top of Instrument

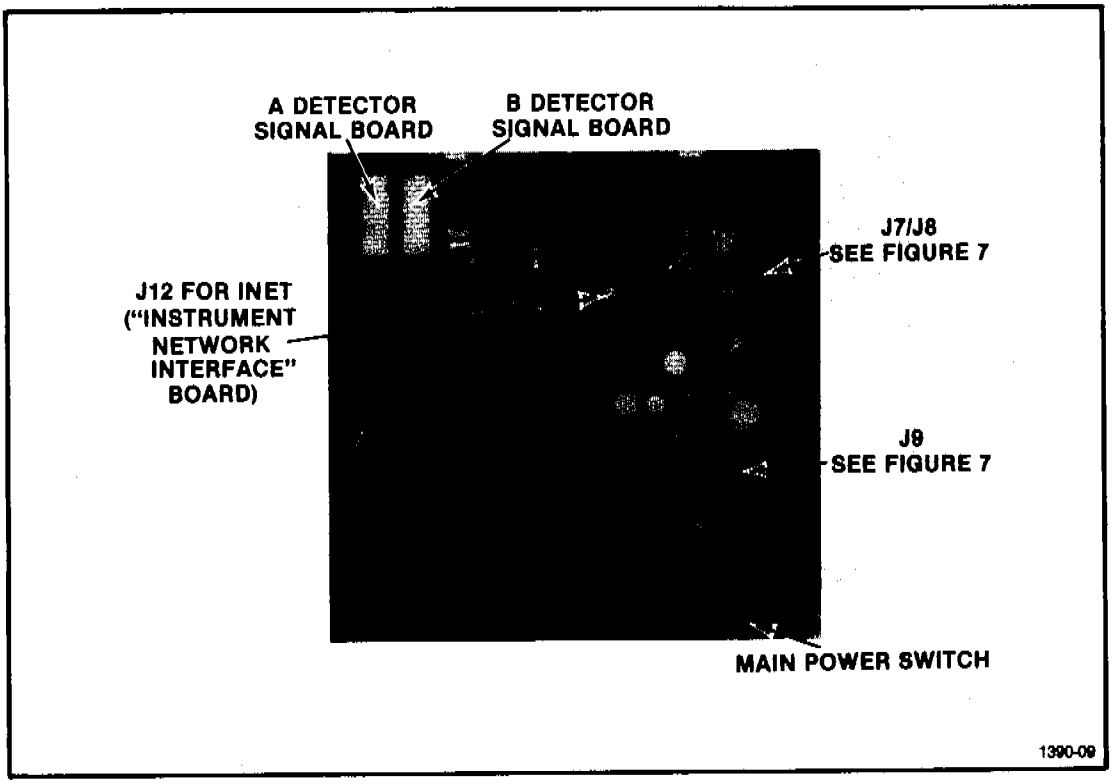


Figure 4. Right Side of Instrument

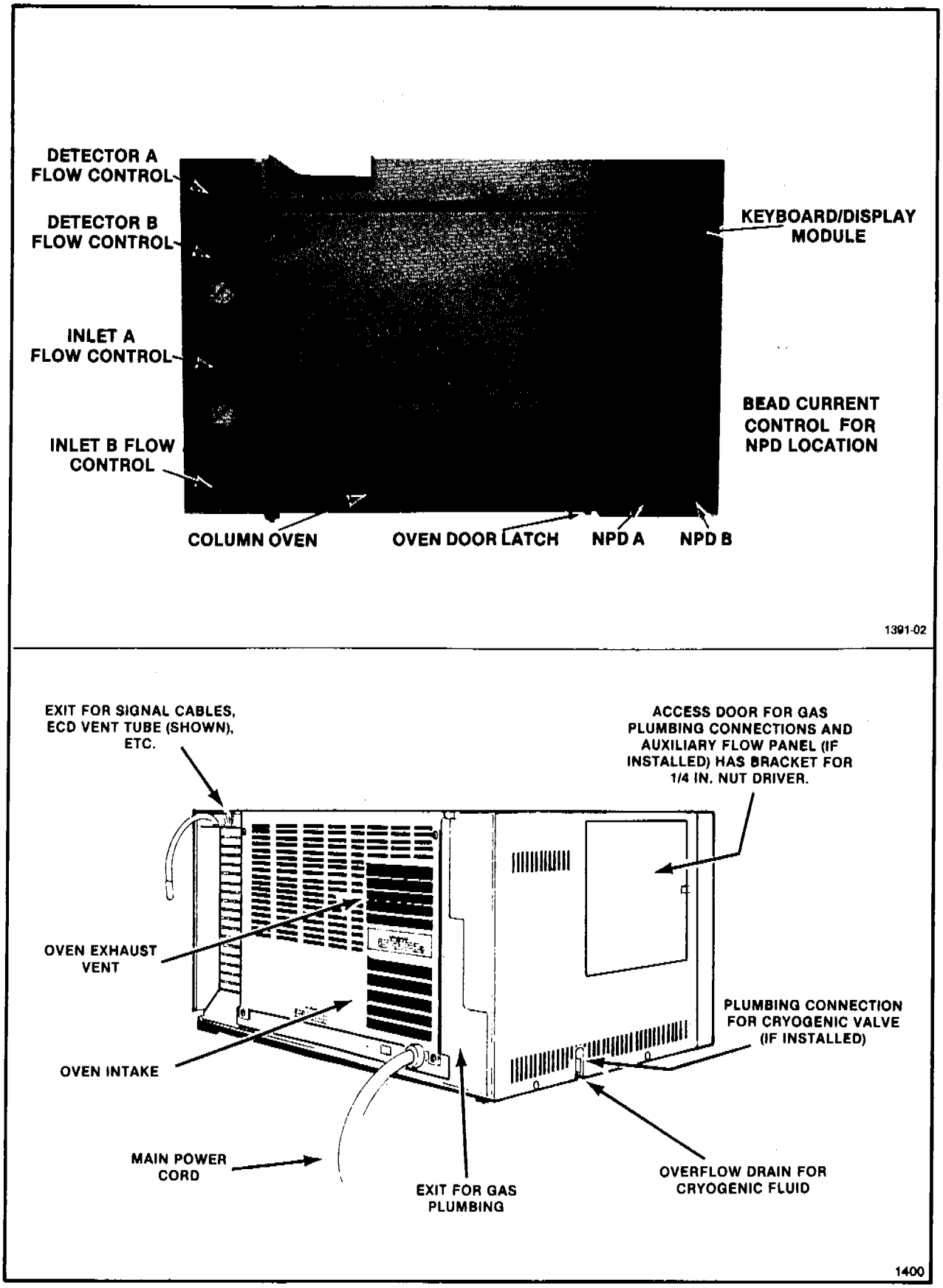


Figure 2. HP 5890A Description

SLOT-DRIVE SCREWDRIVER: 1/8 X 2-inch blade (Part No. 8730-0008). For installing/removing TCD filament and "Delta-T" sensor leads in the signal board connector, and to adjust variable restrictors in detector gas on/off valves.

OPEN-END WRENCH: 7/16 - 9/16 inch (Part No. 8710-0003). For 1/8- and 1/4-inch tube fitting nuts.

COMBINATION WRENCH: 1/4 inch (Part No. 8720-0014). For the capillary column fitting nut.

COMBINATION WRENCH: 5/16 inch (Part No. 8720-0015). For HP (non-swage) flow system fittings (e.g., TCD switching valve tube fittings).

NUT DRIVER: 1/4 inch (Part No. 8720-0002). For FID/NPD jet installation/removal.

NUT DRIVER: 7.0 mm (Part No. 8710-1217). For M4 nuts and thread-forming screws.

HEX-KEY ("Allen") WRENCH: 1.5 mm A/F (Part No. 8710-0909). To tighten NPD transformer lead setscrews.

SPANNER WRENCH: Special (Part No. 19301-00150). For the thermal nut on the FID/NPD base assembly.

INITIAL INSPECTION

Inspect shipping container(s) for damage: if the container is damaged or shows signs of stress, notify both the carrier and your local Hewlett-Packard office. Keep all shipping materials for inspection by the carrier.

Refer to the illustrated parts list included with the specific Accessory installation instructions for parts identification, and for verification of items supplied. If items are missing or damaged notify your local Hewlett-Packard office immediately.

COVER REMOVAL

1. The hinged top cover is removed by bending it enough to disengage a hinge pin, after removing the retaining screws and washers with a 1/4-inch wrench.

2. The area behind the hinged cover is part of the rear panel: remove four screws in the vertical (rear) part of the panel and slide the top part back until it clears the mainframe.

WARNING

HAZARDOUS VOLTAGES ARE PRESENT IN THE MAINFRAME WHEN INSTRUMENT POWER IS CONNECTED. AVOID A POTENTIALLY DANGEROUS SHOCK HAZARD BY DISCONNECTING THE POWER CORD BEFORE REMOVING THE REAR PANEL.

3. The left side panel is secured by two screws at the bottom, and by a molded-in fastener at the top. Remove the two screws and slide the panel back while lifting to disengage the fastener.

4. To remove the top right panel, lift the rear of the panel.

5. To remove the right side panel, first remove the top right panel. Then remove the four side panel screws, two along the top edge, and two along the lower edge, of the panel.

RECOMMENDED HAND TOOLS

Tools listed below are recommended to install Accessories on the HP 5890A. Common tools may be either purchased locally, or ordered from Hewlett-Packard. Any unique tool(s) required to install a particular Accessory (e.g., the 1.5 mm hex key ("Allen") wrench required for NPD installation) are supplied with the Accessory.

POZIDRIVE-TYPE SCREWDRIVER: No. 2 point X 4-inch blade (Part No. 8710-0900). For panel screws, and M4 screws.

POZIDRIVE-TYPE SCREWDRIVER: No. 1 point X 3-inch blade (Part No. 8710-0899). For M3 screws.

SAFETY CONSIDERATIONS

The instrument manual supplied with the HP 5890A, and certain installation instructions furnished with Accessories, contain **WARNING** and **CAUTION** notes detailing potential hazards existing when working on and/or operating the instrument. Before beginning an installation, read and understand these notes.

In addition, the following "common sense" safety considerations should be followed at all times:

1. Hydrogen (H_2) is flammable, and an explosion hazard when confined in an enclosed volume (for example, the oven). In ANY application using H_2, turn off the supply at its source before working on the instrument.

2. The oven, inlet, and/or detector zone(s) may be hot enough to cause burns. Turn off heated zones and allow time for cooling before working on the instrument.

3. To avoid shock hazard, turn off instrument power and disconnect the line power cord from its receptacle any time the rear cover panel must be removed.

4. Wear safety glasses when using compressed gas, and in handling glass or fused silica capillary columns. It is good practice to wear safety glasses at all times when working with the instrument.

5. Also, use care to avoid skin punctures in handling fused silica capillary columns, particularly "HP Series 530µ" columns.

HP 5890A Accessories

Accessories for the HP 5890A are covered by an individual document detailing its installation. This "*Accessory Installation Guide*" contains information applicable to all Accessory installations.

HP 5890A GAS CHROMATOGRAPH

Accessory Installation Guide

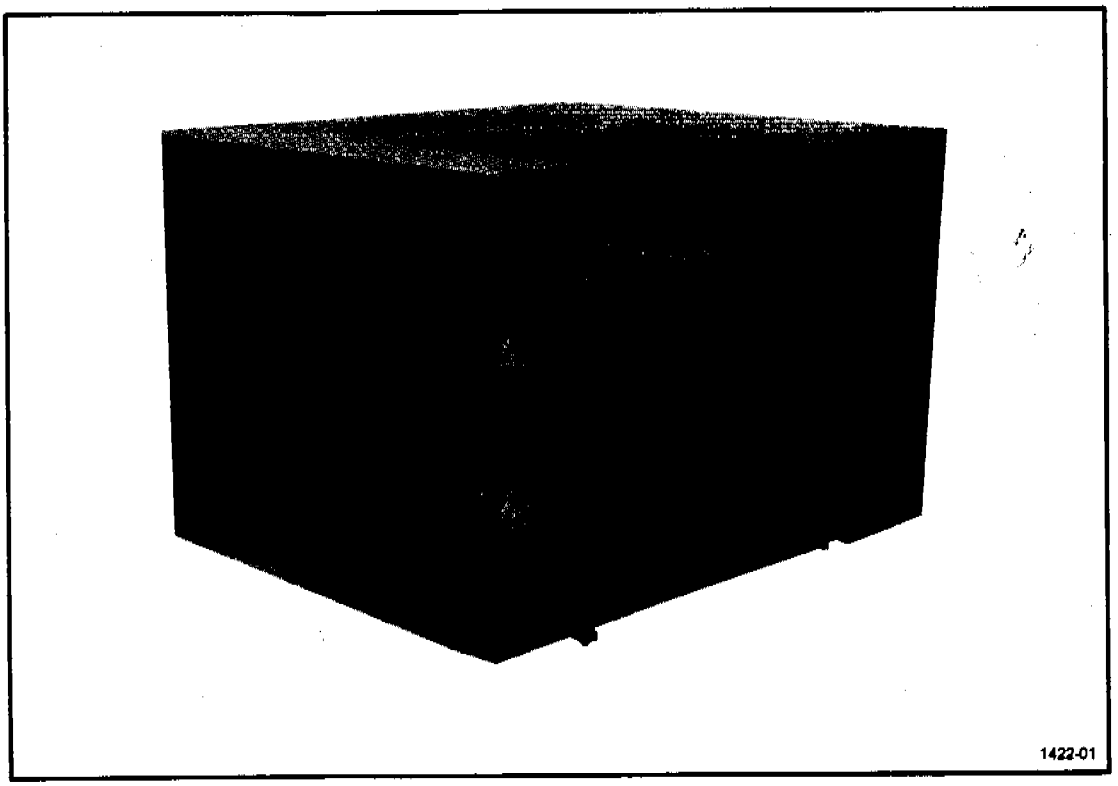


Figure 1. HP 5890A Gas Chromatograph

INTRODUCTION

Accessories are instrument options available for on-site installation to increase or change instrument capability. Once installed, the Accessory becomes an integral part of instrument, and is operated and serviced as directed in the instrument manual.

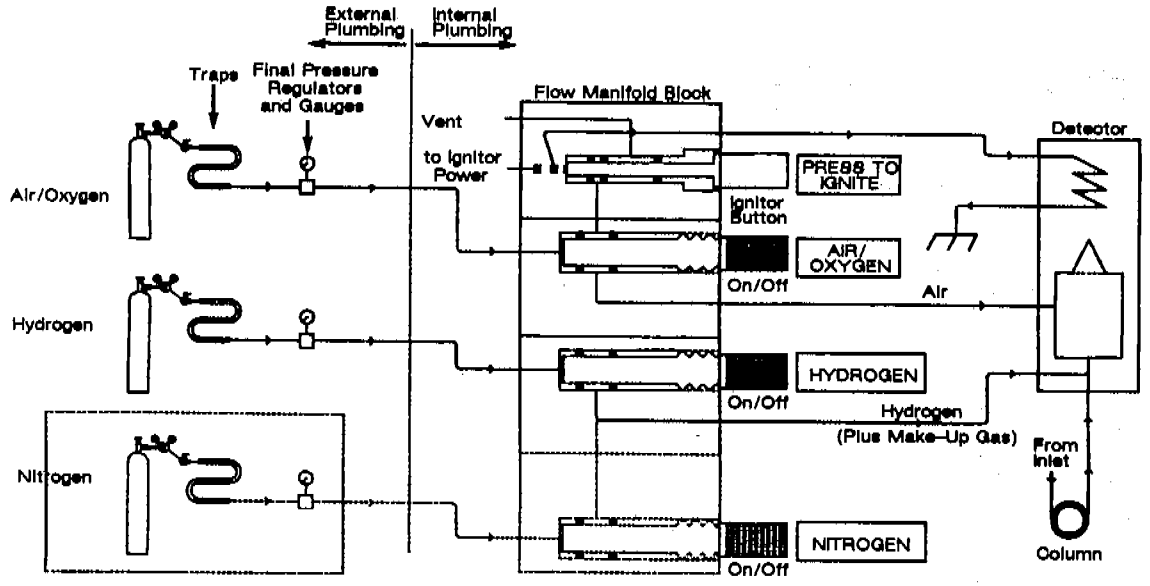
NOTE

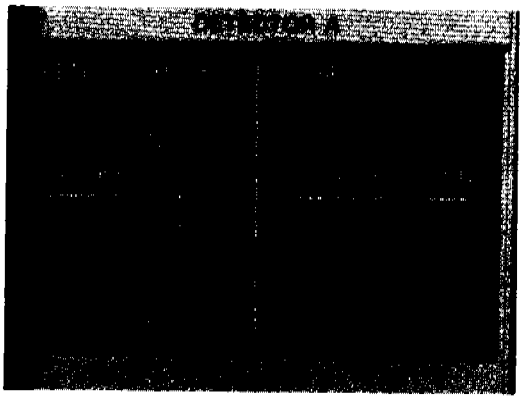
Accessory installation instructions contain information not necessarily available elsewhere: it is suggested this material be filed in *Section 17*, "*HP 5890A Shelf Reference Manual*" for reference.

Part No. 05890-90140
Microfiche No. 05890-98140

(Rev. 3/88)
(1/84) Printed in U.S.A.

PNEUMATIC CIRCUITS





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NOTE

Leak detection fluids (such as "Snoop",etc.) should be used with caution as they could migrate into the flow path and contaminate the system. Alcohol/ water solutions are less likely to cause contamination and may be the better choice for this detector.

FPD Typical Flow Rates

Gas	Flow Rate in ml/min	
	Air Mode	Oxygen Mode
AIR	100	0
HYDROGEN	75	75
CARRIER plus AUXILIARY NITROGEN GAS	15-40	120-140
OXYGEN	0	20

Pressure Check Values

Maximum Pressure: 210 kPa (30 psig)
Pressure Drop: less than 5% per minute

Figure 32. FPD Pneumatic System, Circuit Diagram

FPD HEATER/SENSOR CABLE

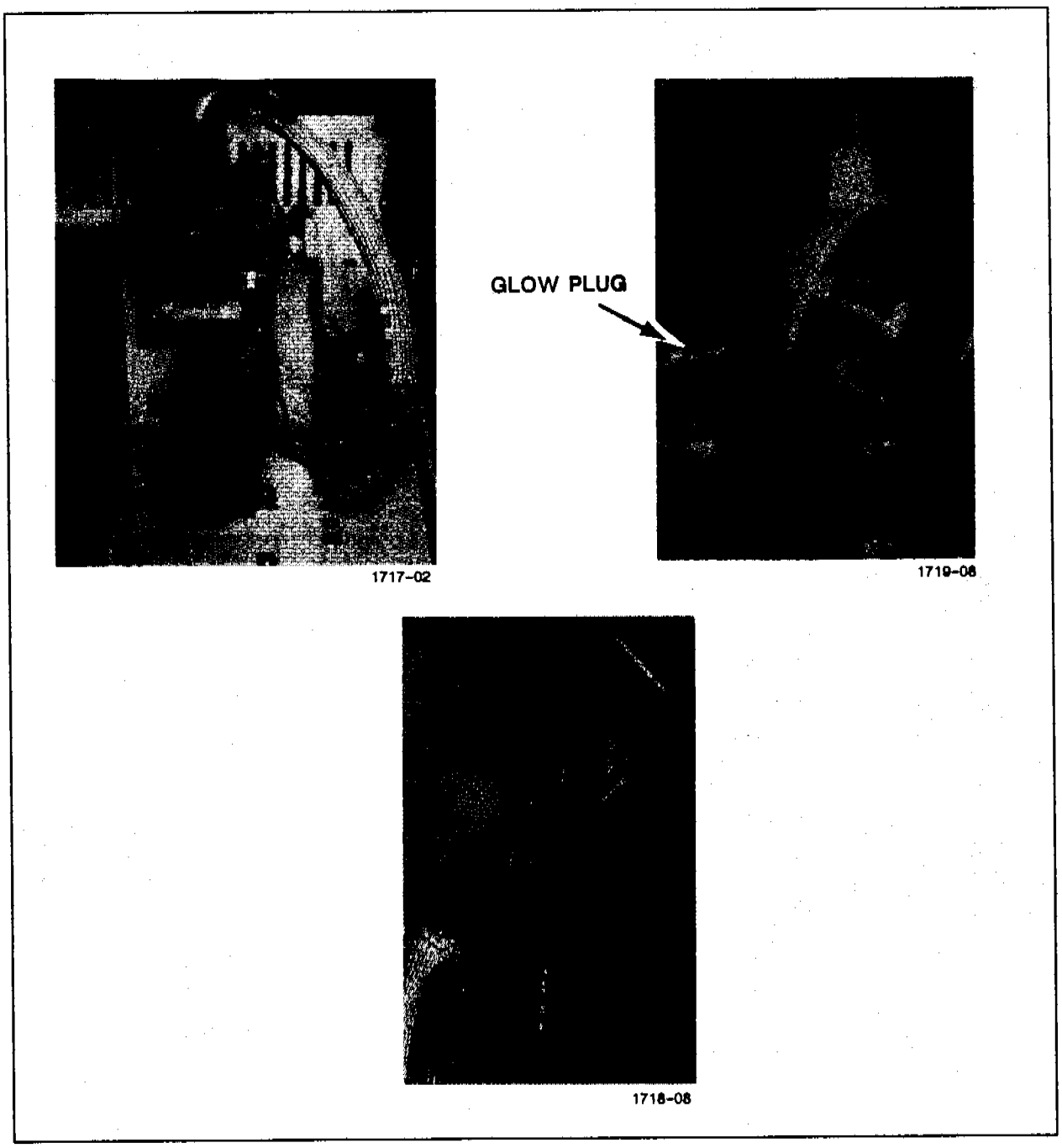


Figure 31. FPD Heater/Sensor Cable Assembly, Part No. 05890-61140

FPD TUBE ASSEMBLY

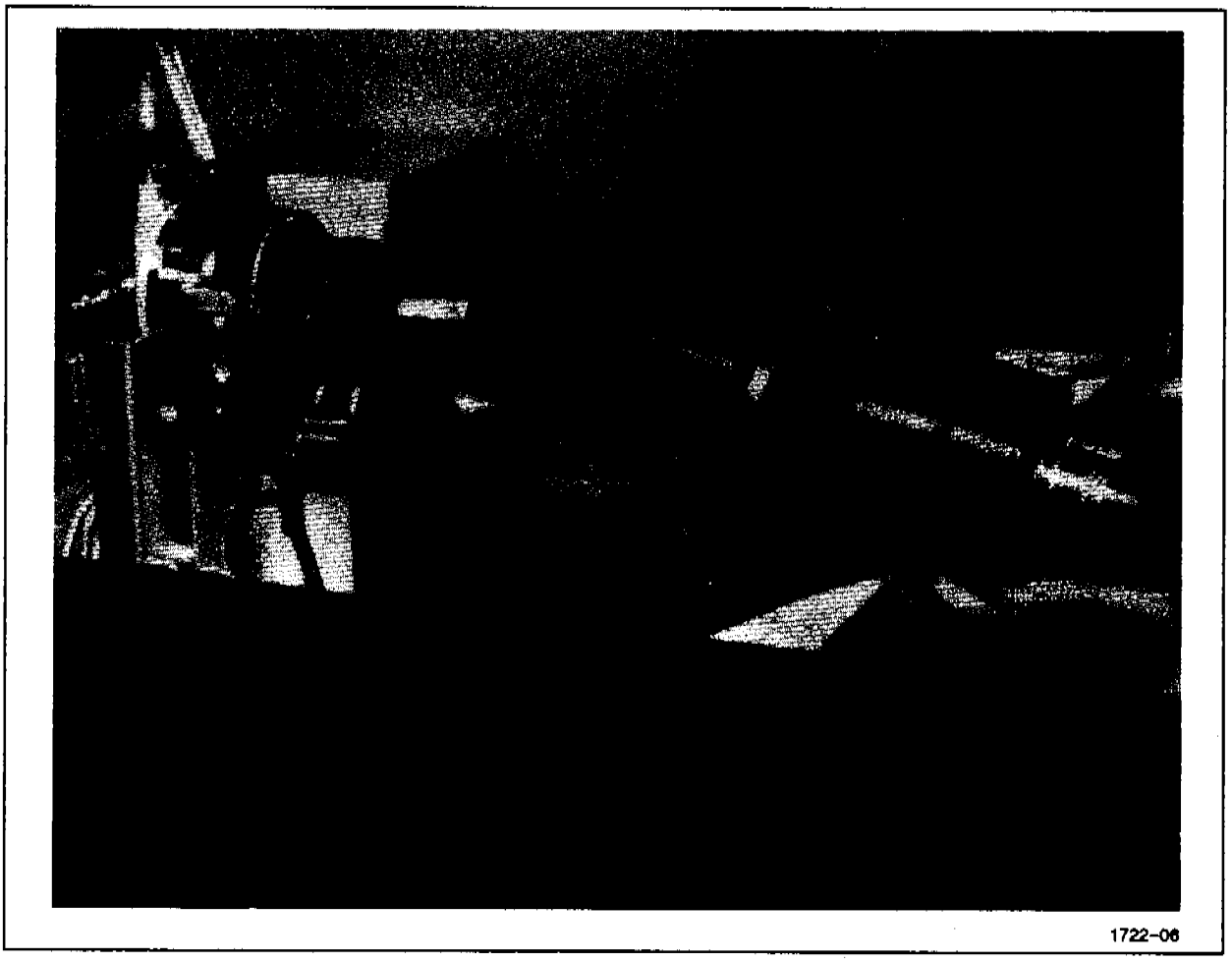


Figure 30. FPD Photomultiplier Tube (PMT) Assembly

FPD PRINTED CIRCUIT BOARD CABLE CONNECTIONS

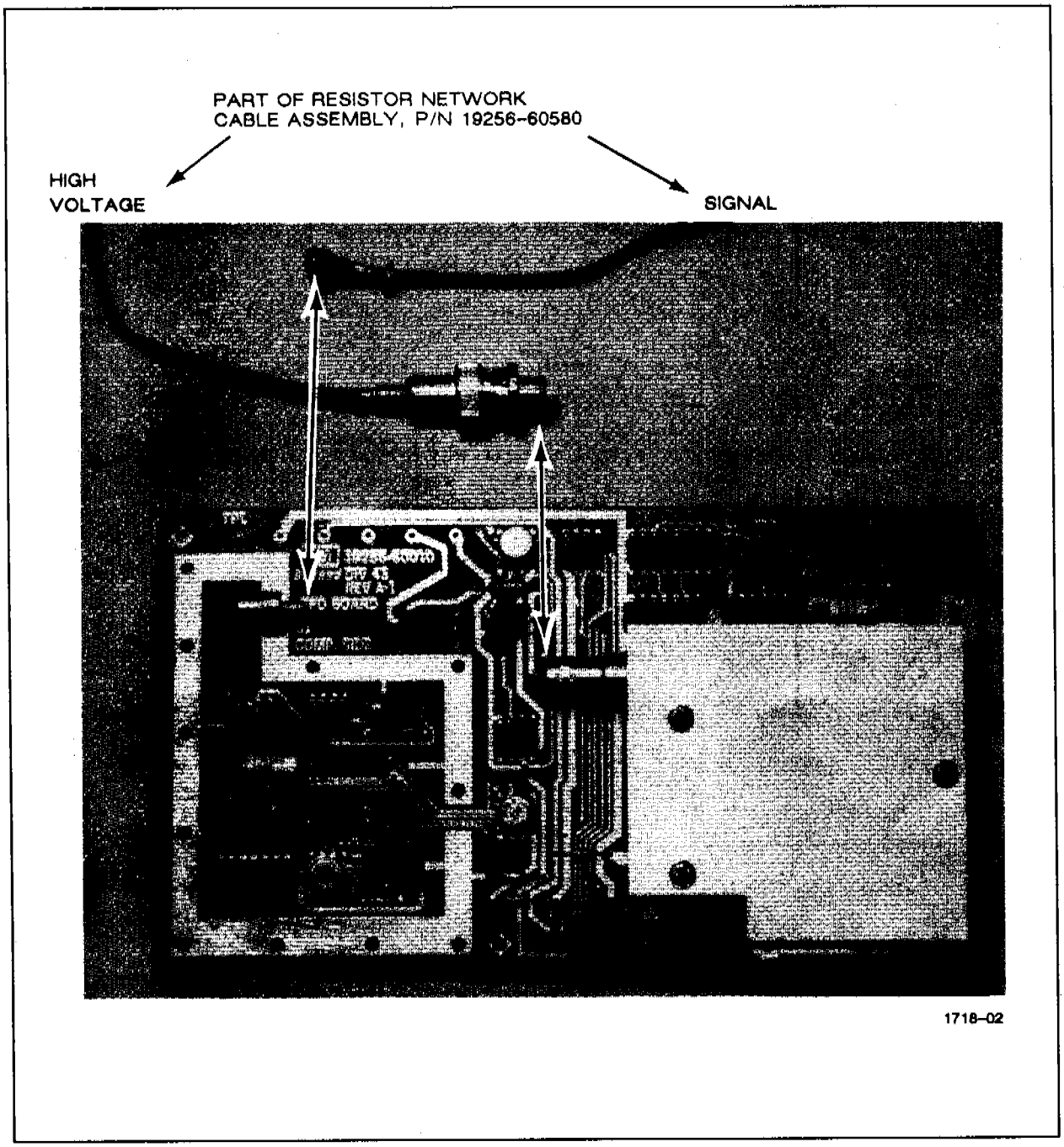


Figure 29. FPD Printed Circuit Board, Part No. 19256-60010

FPD PRINTED CIRCUIT BOARD TESTING AND TROUBLESHOOTING

After installing the FPD assembly and its associated hardware, proper operation of the electronics can be verified as follows:

Conditions for test: flame OFF; all gases OFF; detector "B" OFF.

1. Turn detector "B" OFF;Check test point TP 7 referenced to ground. The voltage reading should be 9.5 +/- 0.5 VDC. Suspect a defective printed circuit board if any voltage does not meet this specification.

2. Turn detector "B" ON; SIGNAL "B" display should read between 140 and 260 counts. Test point TP7 referenced to ground should read between 0.7 VDC and 1.1 VDC. Suspect a defective printed circuit board if count is 0.0 or a light leak if count is above 260.

3. Press and hold the ignitor button, SIGNAL B display should read 65535 (full scale). Suspect a defective resistor network cable assembly or glow plug if display does not read full scale.

WARNING

THE HIGH VOLTAGE (H.V.) CAN BE MEASURED DIRECTLY AT THE HV CABLE OUTPUT AND GROUND, USING A VOLTMETER CAPABLE OF MEASURING VOLTAGES IN EXCESS OF 1000 VOLTS. HOWEVER, THIS METHOD IS NOT RECOMMENDED AS THE POTENTIAL FOR ELECTRICAL SHOCK EXISTS.

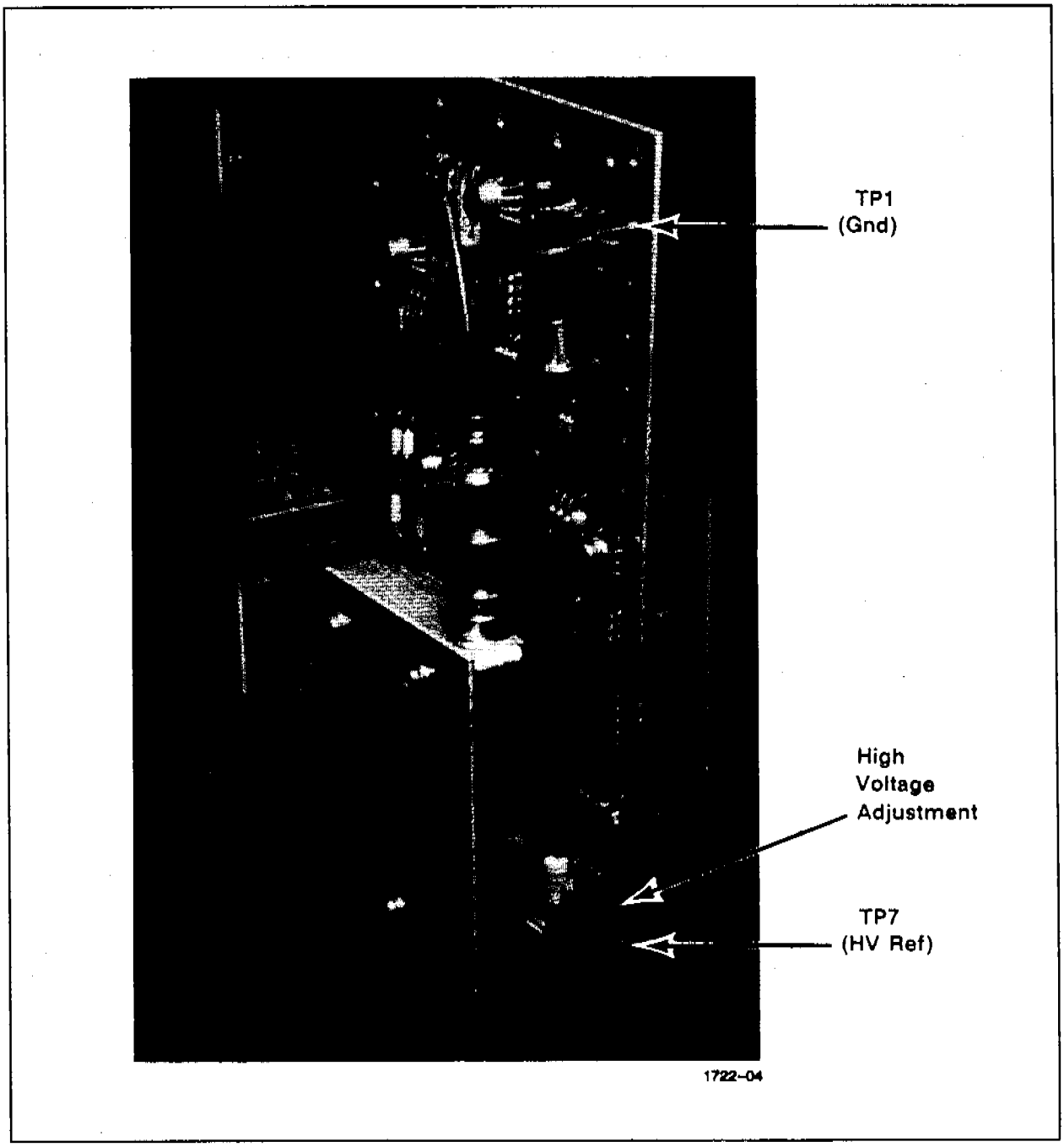


Figure 28. PMT High Voltage Adjustment

HIGH VOLTAGE ADJUSTMENT

High voltage for the photomultiplier tube is set at the factory for optimal sensitivity, however sensitivity may vary from one PMT to another. For this reason, a high voltage adjustment is provided on the FPD signal board for use when a new PMT is installed.

After installing the new PMT, the FPD check-out/performance verification must be performed and results compared with those for the original PMT. Assuming gas flow rates are correct, and the system leak-free, PMT high voltage should be altered only if sensitivity significantly changes.

1. Remove the right side panel to access the detector signal board. The FPD signal board is in the "B" (second) detector signal board slot.

2. On the board, locate the high voltage adjustment and the high voltage reference ("HV REF") test point (TP7).

3. Connect a voltmeter between ground (i.e., TP1 or the aluminum oven top), and HV reference test point (TP7).

4. Set the desired voltage and perform the check-out/performance verification analysis. Pick another voltage and perform the analysis again. Repeat setting the voltage and performing the analysis until maximum sensitivity (greatest area counts for a given amount of sample injected, divided by noise) is attained.

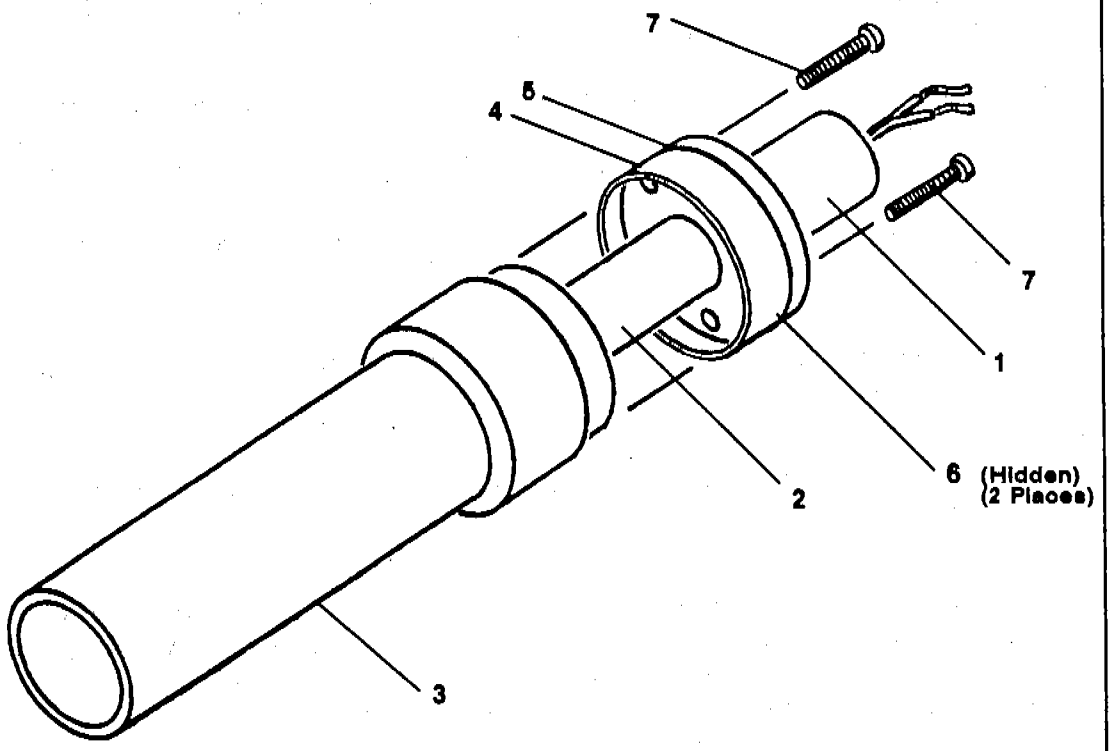
HIGH VOLTAGE SETTING

The voltage set at the factory for optimum sensitivity (signal/noise ratio) is 0.90 +/- .05 VDC (corresponding to -850 VDC +/- 50 VDC at the PMT). The photomultiplier tube (PMT) voltage limits are: 0.72 VDC to 1.05 VDC corresponding to PMT voltage of -670 VDC to -990 VDC.

NOTE

Noise will appear only about 2/3 as large on analog signal paths than on digital, due to high band pass filtering of signal on analog channels.

FPD TUBE ASSEMBLY



Item	Description	Part No.	Qty.
1	Resistor Network Cable Assembly	19256-60580	1
2	Photomultiplier Tube (PMT)	19256-80050	1
3	PMT Tube Body	19256-20650	1
4	End Cap	19256-20710	1
5	Tube Receptacle	19256-20670	1
6	O-ring	0905-1099	2
7	Screw, M4 x 22 mm long	0515-0185	4
8	Tie Wrap (Not Shown)	1400-0308	1

Figure 27. FPD Photomultiplier Tube (PMT) Replacement

REPLACING THE PHOTOMULTIPLIER TUBE ("PMT")

Light from the flame is detected using a very sensitive photomultiplier tube ("PMT"): light strikes a photosensitive surface causing emission of electrons. These are accelerated by high positive potential to the first dynode where several electrons are emitted for each electron striking its surface. These new electrons accelerate towards a second dynode, etc. Electrons "cascade" to the anode which collects all electrons, producing current proportional to the amount of light.

If the PMT is defective (high voltage present and the flame lit: low or no signal and/or high noise not attributed to any other source such as bad cables, light leaks, high temperature, defective signal board, etc.), it must be replaced (Part No. 19256-80050, tube ONLY; Part No. 19256-60510, ENTIRE PMT assembly).

1. Turn off power to the gas chromatograph and disconnect the main power cord.

2. Remove the PMT assembly from the detector module and place it on a clean, suitable work area.

3. Remove the four M4 screws from the PMT end plate retaining the resistor bridge network assembly and remove the end plate. (See figure).

4. Slide the resistor bridge network assembly/PMT out of the PMT housing.

5. Remove the PMT from the socket connector of the resistor bridge network assembly, and replace with a new PMT.

6. Reassemble in the reverse order. Make sure grease, fingerprints, dust, etc. are removed from the PMT window facing the detector module. Also, be sure that both O-rings are in place on the PMT/resistor bridge network assembly, as these are critical light seals. If the O-rings are lost or damaged, replace with part number 0905-1099.

7. Replace the PMT assembly on the detector module.

8. Perform a performance verification analysis using the correct column, sample, sample amount and conditions. If there is a significant change in sensitivity, PMT high voltage may need adjustment (see "High Voltage Adjustment").

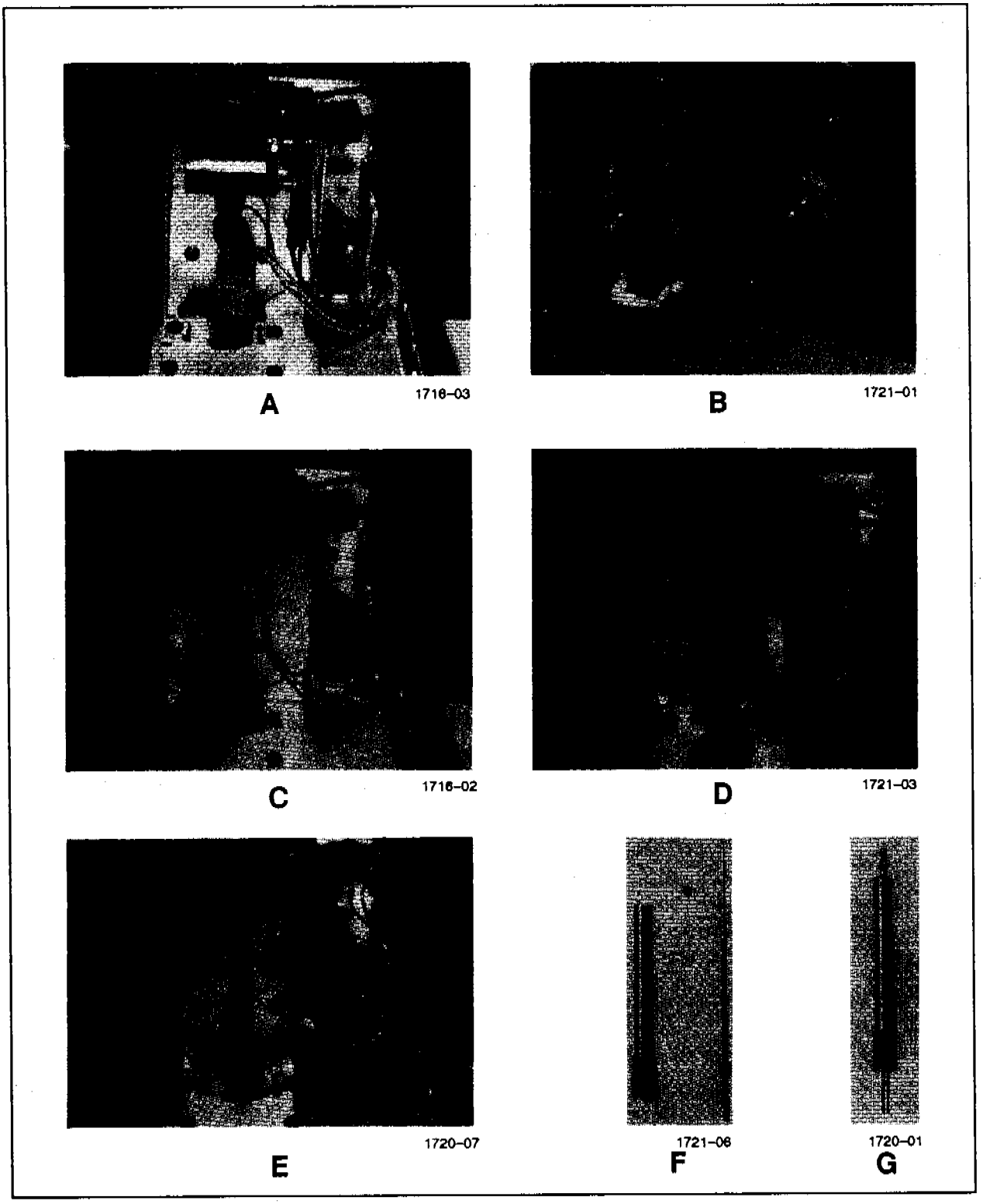


Figure 26. Fused Silica Liner Replacement Procedure

REPLACING THE TRANSFER LINE FUSED SILICA LINER

Occasionally the transfer line fused silica liner between the column and FPD module must be inspected, cleaned, and/or replaced.

1. Remove the FPD burning chamber and jet assembly by following steps 2 through 4:

2. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.

3. Turn off hydrogen, air (or oxygen) and auxillary nitrogen supplies to the detector (manifold on/off valves). For convenience, carrier flow may be left on. Allow time for heated zones to cool to safe temperatures.

4. Remove the photomultiplier tube ("PMT") assembly from the detector module; also remove the filter. Set both in a safe place. Also, remove exhaust tubing (1/4-inch swage fitting) and the chimney assembly (two M4 screws on sides). Then loosen the "U" clamp that locks the burner to support bracket (Part No. 19256-00080), and disconnect the jet assembly from the transfer line tube, carefully lift the detector module vertically from the transfer tube, so as not to damage the fused silica liner. See CAUTION and WARNING notes which follow. Also see Figure 26A, B, C and D.

5. Inside the oven, remove the column to the FPD.

6. Remove the 1/4-inch Vespel ferrule, 1/4-inch Swagelok nut and the lower heated block from the transfer line tube.

7. With a 5/16-inch open end wrench, unscrew the transfer tube weldment from the detector base weldment. Lift the transfer tube weldment (containing the fused silica liner) vertically off the instrument. Remove and inspect the fused silica liner and the 1/16-inch Vespel ferrule by pulling the (fused silica) liner and ferrule out of the weldment from the bottom. Refer to Figure 26E, F and G.

8. If necessary, install a new fused silica liner and Vespel ferrule (combined as part number 19256-60590). When doing so, carefully feed the fused silica liner through the Kalrez O-ring (part number 0905-1101) at the top of the transfer line, so as not to damage the O-ring. Refer to Figure 26F and G.

9. Carefully replace the fused silica liner, ferrule and tube onto the detector base. The fused silica liner should be positioned so that it protrudes approximately 6-7 mm (1/4-inch) above the top of the transfer tube weldment. With a 5/16-inch open end wrench, firmly tighten the transfer tube weldment (1/2-turn past finger tight).

7. Inspect and clean deposits from the jet bore using a suitable wire. If the jet is damaged in any way , it should be replaced. It is good practice to replace the jet, rather than cleaning it, particularly when extremely high sensitivity is required.

8. A new Kalrez O-ring seal (Part No. 0905-1103) must be used when reinstalling the jet into the burner chamber. See section on "Care/Replacement of High Temperature Seals".

9. Reassemble all parts of the detector module; reassemble the module onto the instrument. A new Vespel ferrule (6.4 mm I.D., Part No. 0100-1061) should be used to seal the detector module to the transfer line.

CAUTION

Be careful not to crush or side load the fused silica liner when reinstalling the detector.

WARNING

Whenever working with exposed fused silica tubing, wear eye protection, as fragments of fused silica could be released if the fused silica is fractured or crushed.

10. Reinstall the PMT assembly on the detector module; restore instrument gases and power.

yellow mirror towards PMT

CLEANING/REPLACING THE FPD JET

If a response problem is encountered (sensitivity, noise, selectivity) the FPD jet should be inspected for deposits and, if necessary, cleaned or replaced. To properly service the jet, the detector module should be removed from the instrument, followed by appropriate service:

1. Turn off power to the gas chromatograph and disconnect the main power cord. Remove the detector covers.

2. Turn off hydrogen, air (or oxygen) and auxiliary nitrogen supplies to the detector (manifold on/off valves). For convenience, carrier flow may be left on. Allow time for heated zones to cool to safe temperatures.

3. Remove the photomultiplier tube ("PMT") assembly from the detector module; also remove the filter. Set both in a safe place. Also, remove exhaust tubing (1/4-inch swage fitting) and the chimney assembly (two M4 screws on sides). Then loosen the "U" clamp that locks the burner to support bracket (Part No. 19256-00080). Loosen the swage connection at the bottom of the jet assembly from the transfer line tube, carefully lift the detector module vertically from the transfer tube, so as not to damage the fused silica liner. See CAUTION and WARNING notes which follow.

NOTE

It is unnecessary to disconnect any plumbing, ignitor leads or the heater/sensor. Leave all attached and disconnect the detector block from the transfer line at the 1/4-inch swage fitting, then gently lift block and rotate it enough to access the jet.

4. Remove and inspect the jet assembly. Use a suitable wire or brush to remove any deposits. The jet assembly slips out of the FPD block more easily if the block is still warm.

5. This is also an ideal time to inspect/clean the glow plug (see "Flame Ignition Problems"), and inspect/clean the quartz windows (see "Cleaning/Replacing Windows, Filter, Seals").

6. Use compressed gas, air or N2, to blow out loose particles from the jet and/or detector module body.

CLEANING/REPLACING FPD WINDOWS, FILTERS, SEALS

Column bleed and/or effluent can contaminate the first quartz window (heat shield) nearest the detector module. Dust, fingerprints, atmospheric contaminants can dirty both quartz windows, the filter, and/or the photomultiplier tube ("PMT") window. Contamination anywhere along the light path between flame and PMT can reduce detector sensitivity.

Turn the detector off.

Turn off hydrogen, air (or oxygen), and auxiliary nitrogen supplies to the detector (manifold on–off valves); for convenience, carrier flow and other temperature zones may be left on and at their respective setpoints. Allow time for the detector module to cool.

Remove the PMT assembly from the detector module and remove the filter. Use lint–free lens tissue to carefully clean the filter (both sides), and the PMT window seen inside the housing. Be careful to not scratch surfaces; do not use any cleaning fluid which might leave a film upon drying.

Inspect the filter: chips, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace filter(s) as necessary (sulfur mode filter, Part No. 19256–80000; phosphorus mode filter, Part No. 19256–80010).

Likewise, damage to the PMT window can not be tolerated: if necessary, replace the PMT (see "Photomultiplier Tube (PMT) Replacement").

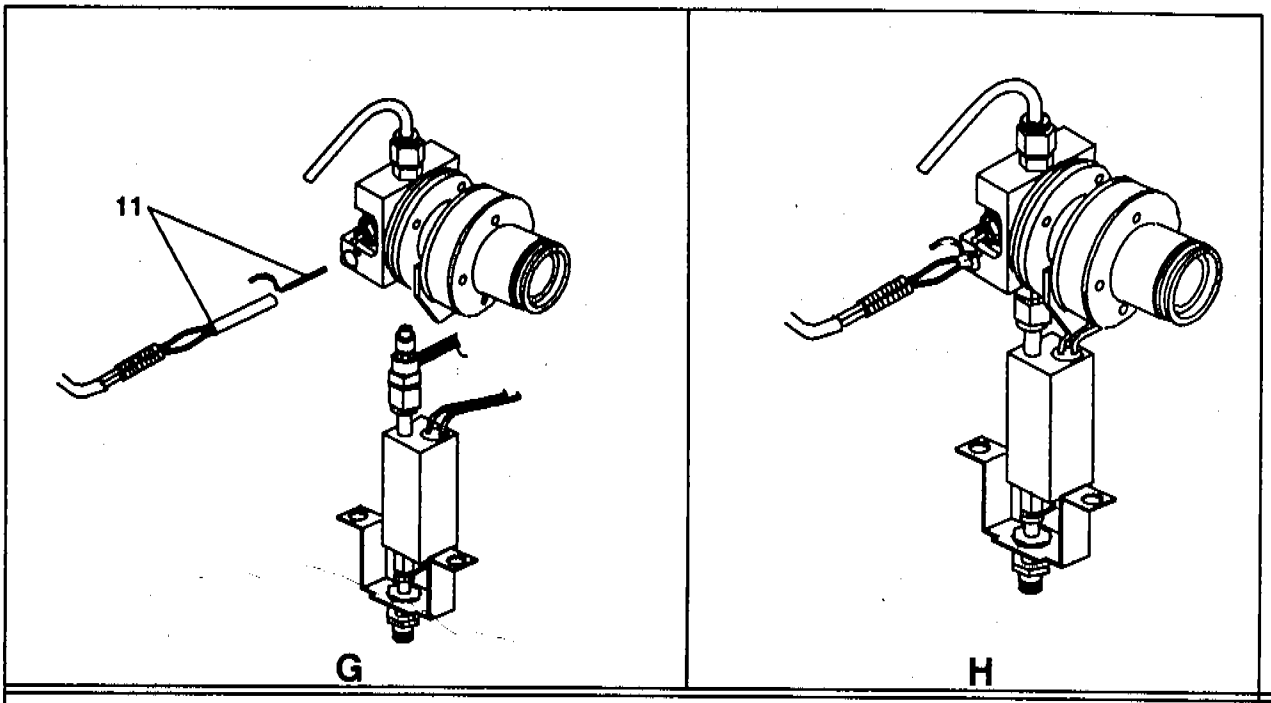
1. Remove four screws to remove the PMT adapter flange. Remove the adapter carefully as a quartz window is exposed and may fall out. window is cleaned in a manner similar to the filter.

2. Remove four more screws to remove the stainless steel coupling. Remove the coupling carefully as the remaining quartz window may fall out. Clean the window in a manner similar to the filter.

Note placement and types of seals found on disassembled parts. Seals should be replaced with new parts on reassembly. (See figure 24 for seal part numbers).

Inspect the windows: chips missing, scratches, and/or cracks in the light path scatter light, reducing detector sensitivity. Replace window(s) if necessary (First window–Part No. 19256–80030; second window –Part No. 19256–80060).

Reassemble parts in reverse order, making sure all seals are of the proper type and in their respective proper locations. Tighten screws evenly and firmly to ensure gas–tight and light–tight seals. If the filter has a silvered side, it should face the flame (indicator arrows > on edge of filter should point toward the PMT).



Item	Description	Part No.	Qty.
1	Weldment, Base	19256-80540	1
2	Gigabore Liner/Ferrule Assembly (see note)	19256-60590	1
3	Lockwasher	2190-0108	4
4	Lower Heater Block	19256-20500	1
5	Weldment, Transfer Tube	19256-80550	1
6	Nut, Brass, 1/4 in.	0100-0056	2
7	Ferrule, Vespel, 1/4 in. ID	0100-1061	2
8	O-ring-Kalrez, Transfer tube	0905-1101	1
9	Weldment, Jet	19256-80580	1
10	O-ring-Kalrez, Jet Cartridge	0905-1103	1
11	Heater/Sensor Assembly	19256-60540	1
12	Spacer, Ignitor	19256-20590	1
13	Glow Plug	0854-0141	1
14	O-ring-Kalrez, Ignitor	0905-1102	1
15	Weldment, Block	19256-80560	1
16	Exit Tube Assembly	19256-20700	1
17	O-ring, Orange, 1.05 in. ID (Silicone)	0905-1104	1
18	Drip Tube, Silicone Rubber	19256-20730	1
19	Gasket, Head Shield	19256-80040	1
20	Window, First Heat Shield	19256-80030	1
21	Disk, Heat Shield	19256-20580	1
22	Coupling, Stainless Steel	19256-20550	1
23	Window, Second Heat Shield	19256-80060	1
24	Screw, M3 x 25 mm long	0515-0065	4
25	O-ring, Orange, 0.926 in. ID (Silicone)	0905-0955	1
26	Flange Adapter	19256-20510	1
27	Screw, M3 x 12 mm long	0515-0911	4
28	Flange Ring	19256-00200	1
29	Clamp	19256-00090	1
30	O-ring, Brown, 1.239 in. ID (Viton)	0905-1100	1

Figure 24. FPD Sub-assembly Parts Identification (Cont'd.)

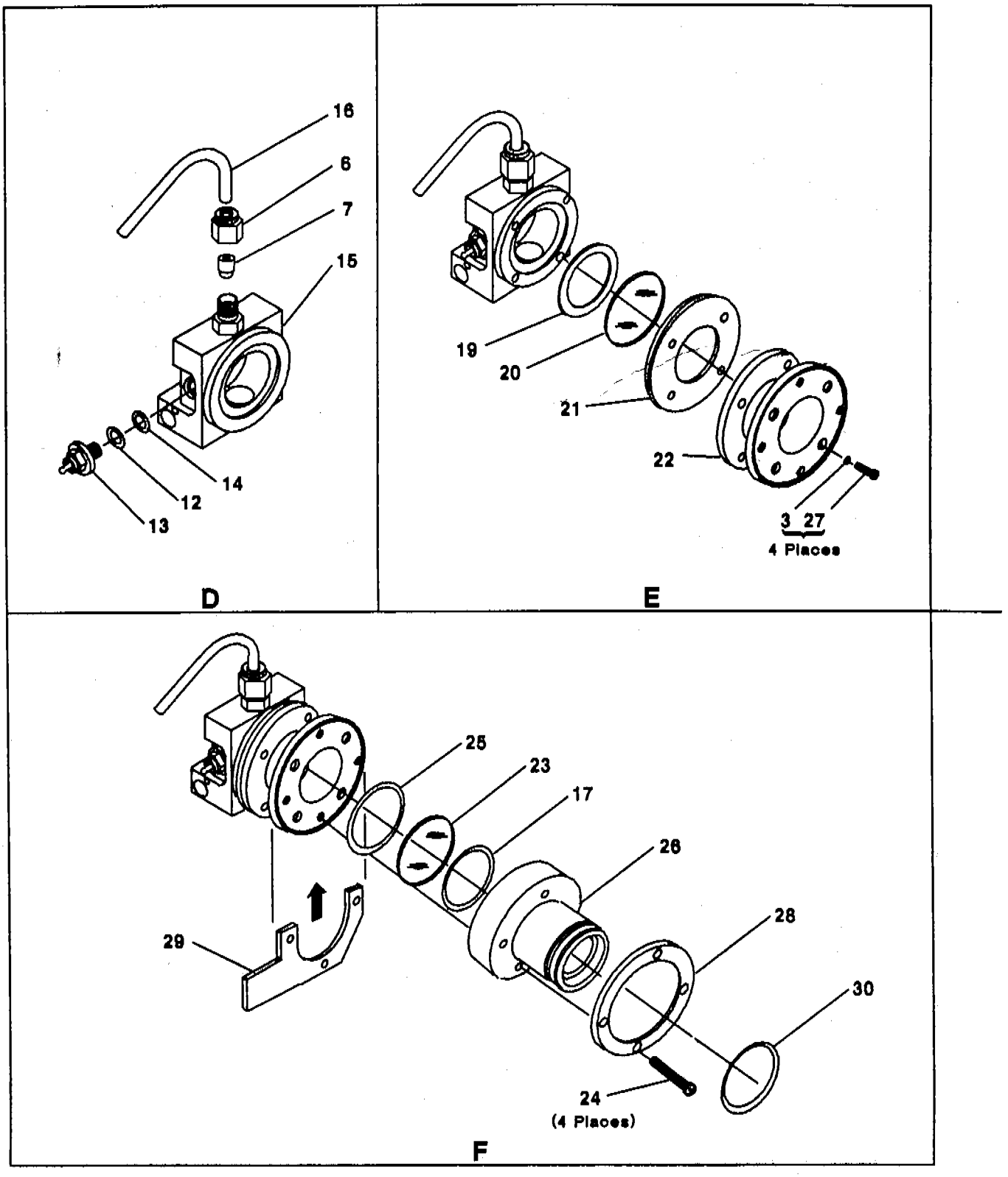


Figure 24. FPD Sub-assembly Parts Identification (Cont'd.)

REPLACEMENT POLICY - MECHANICAL (DETECTOR/PNEUMATICS) FAILURES

Mechanical parts will be replaced at the component or assembly level shown in the related illustration(s).

TOOLS REQUIRED

All of the tools that are required to service the FPD are listed in the INSTALLATION part of this document.

ORDERING INFORMATION

To obtain replacement parts address the order (or inquiry) to the nearest Hewlett Packard Sales and Service Office (see listing at back of this document for addresses). Specify the following information for each part:

1. Model and serial number of the gas chromatograph
2. The Hewlett Packard part number
3. A description of the part

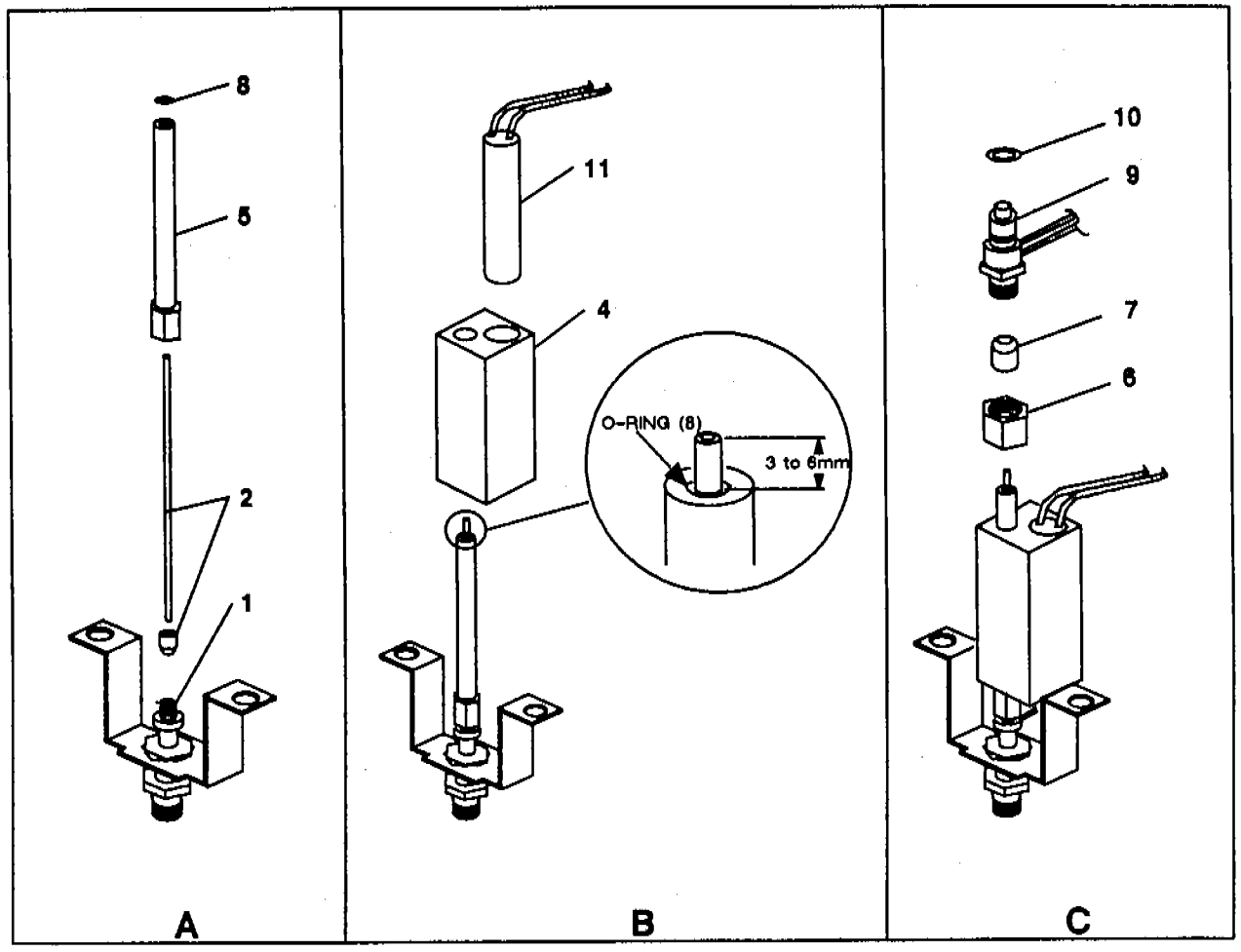


Figure 24. FPD Sub-assembly Parts Identification

Possible leak sources, in order of probability are:

1. septum
2. column fittings
3. supply line swage-type plumbing connections
4. detector block O-ring or Vespel seals
5. other system plumbing.

CAUTION

WHEN LEAK TESTING THE FLOW SYSTEM UNDER PRESSURE, DO NOT EXCEED 210 kPA (30 psig), AS HIGHER PRESSURES MAY DAMAGE THE DETECTOR BLOCK WINDOW OR SEALS.

FPD LEAK TESTING WITHOUT ELECTRONIC FLOW SENSOR

If a leak is suspected in an instrument without an Electronic Flow Sensor, possible leak sources can be checked in the order listed (1 through 5) above. This can be done by capping off the detector exhaust and pressurizing the system to 140 kPa (20 psig). Then close off the supply flow and monitor the rate of pressure drop. Pressure should drop no more than 7 kPa (1 psig) per minute.

WARNING

IF USING LIQUID SOLUTION TO LOCATE LEAKS, TO AVOID A POTENTIAL SHOCK HAZARD PLACE THE MAIN POWER SWITCH IN ITS "OFF" POSITION AND DISCONNECT THE MAIN POWER CORD. BE CAREFUL NOT TO SPILL LEAK SOLUTION ON ELECTRICAL LEADS - ESPECIALLY THE DETECTOR HEATER LEADS!

NOTE

Except when leak testing, it is best to leave the detector module at operating temperature at all times (whether the flame is lit or extinguished) to minimize thermal expansion/contraction.

PREVENTIVE MAINTENANCE

REPLACEMENT POLICY - ELECTRICAL FAILURES

The following electrical/electronic parts will be repaired by replacement only:

1. FPD Printed Circuit Board.

2. Photomultiplier Tube.

3. Resistor Network Cable Assembly.